finishing

NT APPLICATION, ELECTRODEPOSITION, VITREOUS ENAMELLING,
VANIZING, METAL SPRAYING and all METAL FINISHING PROCESSES

Journal

Vol. 3 No. 28 (new series) APRIL, 1957

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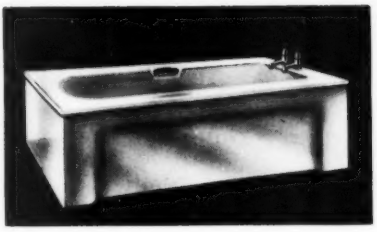

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metal finishing Journal

April, 1957 — AN INDUSTRIAL NEWSPAPERS PUBLICATION — Vol. 3, No. 28 (New Series)

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Telephone: Ripley 136

Published on the 15th day of each month by

INDUSTRIAL NEWSPAPERS LIMITED

Chairman and Joint Managing Director:
Barrington Hooper, C.B.E.
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ADELPHI, LONDON, W.C.2

Telephone: TRAfalgar 6171 (P.B.X.)
Telegrams: Zacatecas, Rand, London

Subscription Terms:
Home and Overseas 24*s.* per annum prepayable
Single copies 2*s.* (2*s.* 4*d.* *post free*)

THIS JOURNAL IS DEVOTED TO THE SCIENCE AND TECHNOLOGY OF PAINT APPLICATION, ELECTRODEPOSITION, VITREOUS ENAMELLING, GALVANIZING, ANODIZING, METAL SPRAYING & ALL METAL FINISHING PROCESSES. THE EDITOR IS PREPARED TO CONSIDER FOR PUBLICATION ANY ARTICLE COMING WITHIN THE PURVIEW OF "METAL FINISHING JOURNAL" AND ALL SUCH ARTICLES ACCEPTED WILL BE PAID FOR AT THE USUAL RATES.

Contents

PAGE

"Organically Sound" **137**

Talking Points **138**
"Platelayer"

METAL FINISHING ANNUAL CONFERENCE ... **139**
Report and Pictures of the Annual Conference of the Institute of Metal Finishing held at Brighton on April 10-13

Personality Spot: R. A. F. Hammond **159**

Overseas Review **177**
"Scrutator"

The Physical Society Exhibition **179**
A Review of prototype and production equipment of metal-finishing interest

Plant, Processes and Equipment **184**

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ORGANICALLY SOUND

IN view of the increasing number of people whose function it is to study, record, analyse and report on widely varying aspects of human behaviour and activity, it cannot be long before a scrutinizing eye is turned on to the present high popularity enjoyed by the Conference. Week after week, from early autumn to late spring, the larger hotels at spas and seaside resorts, which in earlier years would have been swathed in their winter dust sheets, are humming hives of activity as one delegation after another, representing practically every known trade or technical activity, jostle at each others' heels through the revolving doors. Nor, in this scramble for communal discussion, have the summer months remained unscathed, as summer schools and refresher courses have come to fill the gap.

Conferences are going through a period of peak popularity, with most of them attracting record attendances each successive year. Such has certainly been the experience of the Institute of Metal Finishing, whose ninth post-war residential conference was held at Brighton earlier this month. During the three and a half day period of the Conference nearly four hundred members, ladies and visitors took part in the crowded programme.

The Institute of Metal Finishing started its existence over thirty years ago as a band of enthusiastic electroplaters without corporate status under the name of the Electrodepositors' Technical Society. For many years it was concerned exclusively with matters relating to the science and technology of electrodeposition and did much valuable work in giving that very youthful craft a sense of responsibility and technical entity. On becoming incorporated as a company limited by guarantee, a step which was taken some five years ago, the Society adopted a new title, and at the same time assumed, at least by implication, technical sponsorship over a very much wider area of the metal-finishing field.

Initially, however, it is fair to say that the assumption of authority was not followed by any marked efforts to discharge the responsibilities thus undertaken. Changes and developments were taking place at an increasingly rapid rate in the field of paint formulation and application, and the interpretation of these to the users was almost entirely left to the individual trade sponsors of the various new processes and types of equipment.

As a measure of recognition of its responsibilities in the matter the Council of the Institute of Metal Finishing did set up a group—the Organic Finishing Group—within its existing framework, to cater for the interests of what was presumably considered to be a small minority of its members. While it was probably true at that time, entirely as a result of its evolutionary history, that the majority of the existing members of the Institute of Metal Finishing were largely, if not solely, concerned with electrodeposition, there can be no doubt that over the country as a whole, the number of people involved in the application of paint vastly exceeds that of those applying electroplate. Furthermore, it is fair to ask whether, taking the broadest possible view, this arbitrary division of interest is not largely artificial.

The majority of metal articles and surfaces, for reasons that are sufficient and obvious, require the application of a finish. A very large proportion of outworkers, and of finishing departments in large organizations, are equipped and competent to apply a very wide range of finishes. Competition between different types of finish is proper and necessary to ensure that the most effective use is made of them, and that certain finishes are not used where they would be unsatisfactory or uneconomic. However, that is not to say that the whole metal-finishing industry should not have a common platform on which rival finishes should have equal opportunity for their relative techniques and applications to be discussed.

The Group within the Institute of Metal Finishing has shown itself in recent months to be organically sound and serious consideration should now be given to the question as to whether this artificial segregation of interests is not entirely anomalous.

Talking Points by "PLATELAYER"

TOPICAL COMMENT FROM THE MAIN LINES AND SIDE LINES OF METAL FINISHING

A NEW CHROMIUM BATH

ONE of the surprising things about electroplating processes is how little the basic plating baths have changed through the years. Nickel plating solutions (including all the proprietary bright ones) are based on the Watts bath, now 50 years old, while the cyanide silver solution has reached its century literally unchanged. But while these processes have some merit, the chromium plating bath with its fantastically low efficiency, fume and waste-disposal hazards and critical operating conditions has almost nothing to commend it. The obvious way to plate chromium is from the trivalent sulphate bath, but unfortunately smooth bright deposits cannot be obtained from this solution. Numerous addition agents have been tried with varying degrees of success, one of the most important being urea, patented in 1955. This gives bright smooth deposits which, however, are exceedingly brittle.

A more recently announced development is the use of formamide in combination with urea in a chromium sulphate/ammonium sulphate solution which is said to overcome this difficulty. Lead anodes contained in porous pots filled with dilute sulphuric acid are recommended, as anodes immersed directly in the bath cause oxidation to occur. It remains to be seen whether we are on the brink of a revolution in chromium plating practice or whether this is yet another false alarm.

RESEARCH?

WE have, on occasions, referred in these columns to investigations which have shown that the apparently obvious conclusions are not necessarily the correct ones. To this extent, research is warranted into even the most elementary of generally accepted facts. A recent paper on nickel plating, however, seems to go too far in this direction. The authors have worked on the effects of two organic addition agents in Watts type nickel baths which have brightening properties.

After describing the most elaborate experiments involving X-ray and electron diffraction and microscopy studies on the deposits, they reached the conclusion that the increase in brightness caused by the addition agent is accompanied by, if not actually caused by, an increase in surface smoothness and a decrease in crystal or grain size at the surface.

I do not claim to be exceptionally perspicaceous, but my reaction on reading this conclusion is to ask how else can an increase in brightness be caused? If there is no other way in which it can occur (and I can think of none) this result is akin to coming to the conclusion that a knife cuts because the edge is sharp.

This research must have cost someone a lot of money, and one wonders if those paying for it are satisfied.

AGITATING NEWS

HERE is a rather more practical example of a case where the obvious conclusion is not necessarily the correct one. Some recently published work on the cleaning of metals in various alkaline solutions showed that when an oily contaminant containing fatty acids was applied to the metal under perfectly dry conditions, it could be removed from stainless and mild steel surfaces by means of caustic soda solutions, provided the system was not agitated. On agitating the bath the oild was re-deposited and not removed when the agitation was stopped. If, however, the soil was applied under moist conditions the caustic soda was ineffective in completely removing it. This, of course, is only part of the story and a good amny other findings were reported on the effects of different a'kalis.

The point of my present comment is that most people would expect that any kind of soil would be removed more easily in any kind of vigorously agitated alkaline solution than in a still one. One never knows till one tries.

DON'T ALL RUSH AT ONCE!

PLATERS who are being harassed by local authorities about their effluent disposal arrangements will find in Alderman Johnson of Malden, a man after their own hearts. The Alderman is Chairman of the Finance Committee of the local Council, which is now being asked to join in constructing a large new sewage plant with two other adjacent authorities. Commenting on the cost of the scheme, the Alderman is reported to have said:

"We have run our disposal works quite happily for many years without a chemist on the staff, but now there will be three chemists and one laboratory assistant. Why they want all those chemists I really do not know. Their job is to dispose of the stuff, not to test it, smell it, and fool about with it."

THE METAL FINISHING CONFERENCE

A Report of the Annual Conference of the INSTITUTE OF METAL FINISHING held in Brighton on April 10-13

THE ninth in the series of major residential Conferences organized by the Institute of Metal Finishing since the end of the war was held at the Grand Hotel, Brighton, on April 10 to 13 this year.

The metal-finishing industry, or, more particularly, that section of it concerned with the electrodeposition of metals, has in recent years accorded considerable and increasing support to these annual occasions, and the number of delegates attending them has shown a steady growth. Once again, this year, the number of registered delegates constituted a record, and the attendance at the culminating Conference Dinner was the largest yet achieved at this function.

While the major emphasis of the technical programme lay in the field of electrodeposition, the growing strength of the Organic Finishing Group within the Institute was reflected in the fact that two of the six technical sessions were devoted to the discussion of papers relating to paints and their application. It was apparent from the attendance at these two sessions that the alleged division of interest among members between the two fields is largely artificial, and that a large proportion of those attending the Conference were interested in all aspects of metal finishing as a subject.

The official proceedings of the Conference were launched on the first morning by a formal address of welcome from the Mayor of Brighton, who was received by the president of the Institute, Mr. R. A. F. Hammond, and the president-elect Dr. T. P. Hoar. On the same evening delegates and their ladies were the guests of the Mayor and Corporation at a civic reception and dance at the Hotel Metropole.

The principal social event of the Conference was the Conference dinner and dance held on the final evening at the Grand Hotel. This attracted a capacity attendance of very nearly 400 members with their ladies and guests. Principal speaker at this function was Sir Anthony Bowlby, Bt., who proposed the toast of the Institute to which the president replied. The toast of the Guests was proposed by Dr. G. L. J. Bailey and responded to by Dr. L. F. C. Northcott.

On Saturday morning a rather smaller number of members than usual took part in the annual golf match for the John Preston Trophy. Winner this year was Mr. R. G. (Jim) Hughes, with Mr. A. K.

Jordan and Mr. G. Smallwood, second and third.

During the Conference provision was made, by kind invitation of the directors of the companies concerned, for members to visit the works of either Underwood Business Machines Ltd., or Allen West and Co. Ltd. Those who participated in these visits were enabled to see most interesting examples of modern high-speed production methods into which a number of finishing processes had been integrated.

The Conference was also used as the occasion for the presentation of the 4th Hothersall Memorial Lecture by Dr. V. D. Evans. A large audience listened to Dr. Evans, who dealt with certain aspects of corrosion in relation to nickel plating and delivered his remarks in his own fascinating and individual style.

The opportunity was taken during the Conference Dinner to present the I.M.F. Gold Medal, awarded for distinguished services to the Institute and the industry, to Dr. G. E. Gardam. As a past-president and a founder-member of the Electrodepositors' Technical Society from which the Institute of Metal Finishing evolved, Dr. Gardam had worked indefatigably in the interests of the Institute and the award of the medal was warmly acclaimed by the assembled members.

The majority of the papers presented to the technical sessions related to the results of research and investigation into aspects of electroplating, and in one instance a paper described, for the first time in this country, a novel process for the deposition of metals from powder by peening. In this process, which is particularly suitable for the bulk coating of large numbers of small components, the articles to be processed are enclosed after cleaning in a tumbling barrel with a carefully measured amount of the appropriate metal powder, a specific tumbling medium, and a special addition agent or promoter. The coatings produced after some 60 minutes tumbling are very adherent and closely controllable as to thickness, with excellent throw into all areas accessible to the medium.

Abstract No. 1:

An Investigation of the Mechanism of Levelling in Electrodeposition

By S. A. Watson, B.Sc., A.R.I.C. and J. Edwards, Ph.D., B.Sc.†*

AN electroplating solution exhibits levelling action if it yields deposits which are smoother than the surfaces to which they are applied. It is suggested that solutions exhibiting levelling action contain a minor constituent (usually added deliberately as a levelling agent) which is incorporated in the deposit and causes a fall in the deposition potential. Under suitable conditions the removal of this levelling agent from the solution by adsorption or co-deposition on the cathode, leads to the establishment of a diffusion layer with a reduced concentration at the cathode surface. The rate of diffusion of the levelling agent from the main body of the solution is much greater to the peaks than to the recesses of a rough surface: the "ideal" distribution of diffusion rates, assuming uniform concentration at the surface, would be the same as the primary current distribution. Consequently, more levelling agent is incorporated in the deposit and the local cathode potential is correspondingly lower on peaks than in recesses. Current is thereby diverted to the hollows and the surface is progressively levelled.

In support of this hypothesis the authors show that:

1. Levelling agents lower the cathode potential.
2. They are incorporated in the deposit.
3. The extent of the incorporation is greater on the projections than in the hollows of a rough surface.
4. The incorporation of the levelling agent in the deposit leads rapidly to the establishment of a diffusion layer.
5. A curve relating levelling power to concentration of levelling agent can, on the basis of this hypothesis, be calculated from curves relating cathode potential to concentration and current density, the theoretical and experimentally determined curves showing many points of similarity.

Tests samples were cut from Decca and E.M.I. electro-formed copper record matrices, and cathode efficiency was determined on polished copper cathodes which were weighed before and after plating. A special plating cell was designed and used in order to obtain reproducible results in the measurement of levelling. In addition to levelling power, measurement was also made of cathode potential and a determination made of the distribution of incorporated matter in the deposits. A wide range of plating solutions and addition agents were used in the experiments and the effects of varying current density, surface condition, etc., etc., were also measured.

As a result of the experiments carried out it has been concluded that under suitable conditions, substances which, when added to an electroplating solution, are incorporated in the deposit and lower the cathode potential, *i.e.*, make it more negative, should behave as levelling agents.

If a single addition is used, the levelling power is critically dependent on concentration which should therefore be carefully controlled. This applies whether the levelling-concentration curve shows a maximum or continues to rise up to the point at which the properties of the deposit become

* *Investigator in Electrodeposition Section, and*
† *Head of Electrodeposition Section, British Non-Ferrous Metals Research Association, London.*

inadequate. It is also possible to calculate approximately from known relationships between cathode potential, levelling-agent concentration and current density the way in which levelling power varies with changes in plating conditions.

In choosing the appropriate concentration of levelling agent it should be remembered that the current density varies, often widely, over most articles which are electroplated. It might be desirable, therefore, to choose a concentration of levelling agent which would give maximum levelling power at a current density lower than that at which the plating solution is to be used, thus ensuring good levelling power where the plate is thinnest.

The use of more than one levelling agent might offer an alternative way of obtaining the best overall result on shaped articles, but further experimental work would be required to establish this completely. Agitation of the solution would be expected to have little effect on the maximum levelling power attainable but would probably be necessary in most cases to maintain adequate circulation and prevent plating defects.

Factors which increase the slope of the polarization/current-density curve, *e.g.*, complexing of the principal metal ions, tend to make the current density more uniform, therefore, although they increase throwing power, they would decrease levelling power.

An increase in the conductivity of the plating solution would increase levelling power as well as throwing power, but the effect on levelling power is likely to be relatively small. Furthermore, levelling power increases as the scale of roughness decreases but the effect becomes progressively smaller. Finally, it is concluded that there is no theoretical limit to the levelling action which can be attained.

Abstract No. 2:

The Influence of Periodic Reversal of Current Upon the Surface Roughness of Electrodeposited Copper

By A. Hickling, D.Sc., Ph.D., F.R.I.C. and H. P. Rothbaum, M.Sc., Ph.D.**

THIS paper reports the results of experimental work which show that under suitable conditions periodic-reverse plating from copper-cyanide solutions gives a surface whose roughness, after a short initial period, is independent of the length of plating time, and of the initial state of the surface, whether rougher or smoother than this plateau value. The plateau height is dependent upon the ratio of cathodic to anodic quantity of electricity and it decreases as this ratio is diminished, and it can also be lowered by agitation. Most of the work, which was carried out in a specially designed cell, was done without any addition agent present, but in a few experiments potassium thiocyanate was added and this also seemed to decrease the roughness value of the plateau. In contrast, electroplating from copper-sulphate solutions is not appreciably affected by P.R.; in fact the rate of roughening with d.c. is much slower than with cyanide electrolytes, but the roughness in both d.c. and P.R. is directly proportional to the effective plating time and no roughness plateau is attained. Essentially similar results were found in some experiments using copper-phosphate solutions.

With cyanide solutions the initial rate of roughening is much slower than it subsequently becomes and if the duration of cathodic deposition can be kept within this kind of period of induction then, as has been shown by direct experiment, specimens roughen relatively slowly. With the P.R. cycles normally employed this condition is always satisfied.

Rough copper surfaces anodically treated in cyanide solutions undergo marked smoothing. Thus in P.R. electrolysis it may be expected that an initially smooth surface will roughen up to the point at which roughening in the cathodic pulse and the smoothing in the anodic pulse are equal when no further change should occur. Similarly an initially rough surface should smooth to the point at which the two opposing tendencies are equated and this has been found in the occurrence of the plateau of roughness in P.R. deposition. Any factor tending to increase the smoothing relative to the roughening should decrease the height of the plateau, and this is most clearly indicated in the variation of anodic/cathodic quantities of electricity. Any reduction in the rate of roughening *e.g.*, by agitation or by the use of an addition agent, should decrease the final roughness and this agrees with the results of the experiments.

Although it has often been suggested that the effect of P.R. is due to a combination of electropolishing and electrodeposition, this is not so, as with P.R. the electrode does not normally enter the electropolishing region; this has been confirmed by the experiments. In fact the smoothing which occurs during the anodic part of the cycle seems to be of the type which can occur during etching, which precedes the electropolishing region. However, during the anodic pulse in P.R. electropolishing can occur under special conditions. Further to clarify this aspect a special set of experiments was carried out with an electrolyte of copper sulphate in phosphoric acid, but using ordinary P.R. cycles and currents the electrode did not enter the polishing region, and, in addition, P.R. was found to have no appreciable influence upon the general roughness of the deposit, although edge roughness was reduced. However, by fixing the anode potential such that the specimen was bound to enter the polishing region in each anodic

* *Department of Physical Chemistry, Liverpool University.*

pulse, gleaming bright electrodes could be obtained, with surfaces free from fine irregularities, but showing considerable waviness. The normal P.R. surface is one of ordinary but restrained roughness.

Copper deposits from cyanide solutions are extremely fine grained and it seems not unlikely that in this case the development of surface roughness is very largely dependent upon polarization conditions at the metal/solution interface. Should roughness once commence, *e.g.*, from intrinsic roughness in the basis metal or by blocking of positions of the surface by hydrogen, there are bound to be inequalities in the concentrations of metal-bearing ions near the surface. Quite apart from the differences in distance of projecting points on the cathode from the anode, peaks in the surface will be more accessible to diffusing ions and will tend to receive more current and grow progressively. This tendency will be reinforced in the case of cyanide solutions by the large differences in potential which accompany small changes in the concentration of dissolved copper. If over the surface of a cathode in cyanide solutions there is a variation of concentration of the copper-bearing ion, the points at which the concentration is highest will receive a predominant portion of the current passing. Such points, however, are already tending to grow more rapidly than the remainder and thus the effect is progressively amplified. It is likely that this accounts for the rapidly accelerating roughening in d.c. deposition from cyanide solutions.

P.R. electrolysis can restrain development in two ways: first, it is very effective in minimizing concentration polarization and maintaining uniform concentration of metal-bearing ions over the surface, and, second, dissolution during the anodic part of the cycle is constantly dissolving the peaks in the surface more rapidly than the hollows and minimising the irregularities formed. The fact that P.R. has little influence on general roughness in the deposition of copper from sulphate and phosphate solutions is probably due to this roughness arising in a different way. It has been shown, for example, that the deposits are made up of fairly large crystals and the roughening, which is much slower than in cyanide solutions, arises probably by a process of crystal growth. In sulphate solutions anodic dissolution does not seem to produce the same degree of smoothing that it can do in the cyanide electrolyte. Thus, it is suggested that the very marked effect of P.R. in restraining roughening in the deposition of copper from cyanide solutions may be largely specific to this particular case.

Discussion

Dr. T. P. Hoar (University of Cambridge) said that in the paper by Watson and Edwards the authors had made an excellent case for the very elegant mechanism which they had proposed. One of the interesting highlights in the paper was the action of thiourea, or, more properly, the dual action of thiourea. The authors had shown very conclusively that thiourea in small concentrations produced roughness, and, far from increasing the cathode polarization, it actually reduced it. Those two facts formed a very convincing confirmation of the theory; the increase of cathode polarization produced roughening instead of levelling.

When very small quantities of thiourea and its derivatives are used as inhibitors of the acid attack on steel or nickel, then anodic stimulation, not inhibition is produced. Could the authors suggest any kind of mechanism by which thiourea could be stimulative in small quantities and inhibitive in large?

The position of the maximum degree of levelling was of considerable practical importance, because if a levelling agent is used then it is required to produce the maximum possible effect, and the maxima as shown in Figs. 11 and 12 of the paper were rather sharp. Was the maximum affected by various factors in the system? Dr. Hoare thought that it was affected, in particular by the size of the discontinuity. He expected that with a much larger groove it would be shifted to a larger concentration of the levelling agent. Consequently, depending on the amount of levelling aimed for, *e.g.*, the filling of large cracks on the one hand, or merely producing a very good surface on a somewhat irregular one on the other, it might be necessary to use a quite different optimum quantity of levelling agent. The authors pointed out that the maximum occurred at a different concentration for different agents, and that must be reflected in the diffusibility or diffusion constant of the levelling agent in solution, and possibly the different rates of adsorption or de-adsorption of that levelling agent.

Such factors would also be affected by temperature, and indirectly the temperature might be expected to have a considerable effect on the precise quantitive operation of any of the levelling agents. The authors had suggested that the use of more than one levelling agent might be a solution of this problem of the rather sharp maximum in that with two superimposed levelling agents there might be a flattening out of the curve of levelling power against concentration, and therefore a rather wider range.

Effect of Cadmium

The paper did not give any results for the interesting case of cadmium as an agent. Dr. Hoare believed that the authors used cadmium in rather small quantities, about 0·5 gm. per l., and they showed that the presence of cadmium in solution tended to give cadmium on the peaks and less or no cadmium in the hollows. He wondered

on what grounds the authors took the blackening of the peaks as evidence of cadmium in the deposit? He said this because cadmium sulphide is yellow in colour. Was what they saw actually an interference colour, caused by an interference film, which should be a dark blue colour, of calcium sulphide, or was there a considerable amount of material, in which case he would have thought that it would be a yellow colour?

Finally, what about metals other than cadmium as possible levelling agents for nickel? The nickel-cobalt bath was well known to give fairly bright deposits, and this diffusion limitation which the authors showed in this case at ordinary current densities would not be expected. Was it possible that other metals, rather like nickel, other than cadmium, might be promising or possible addition agents to provide cathodic levelling?

Dr. D. N. LAYTON (Ionic Plating Co. Ltd., Birmingham) said that he had been carrying out a fair amount of experimental work with P.R. plating in an industrial sense, and Dr. Hickling and Dr. Rothbaum had done nearly all the experiments which he would like to have done if he had had the time. His own observations of the mechanism of P.R. plating bath supported everything that these authors had put forward.

Electropolishing Effect

On the question of electropolishing during the anodic part of the cycle, he was sure that Jernstedt and the present authors were quite correct in saying that electropolishing was not an essential part of the operation. There were various reasons for saying this, one being that the authors had found that it was possible to get electropolishing in the cyanide solution provided potassium hydroxide was absent. In the bath normally used there was a considerable quantity of this. In practical plating with P.R. the surface blackened within a very short time at the current densities normally used, which were not much less than that used by the authors, and after a period of 4 sec. the surface was black.

From the authors' results it might be thought that any further period of time anodic would be wasted, but his view, on the basis of practical experience, was the contrary. In other words, if the current was reversed to allow the surface to go black there was not quite the same degree of levelling as if the specimen was left anodic for a longer period of time. Polishing did not occur during that period and if the black film was removed it would be found that the surface is dull, matt.

CONFERENCE CAMEOS

(Top, left to right) Mr. R. A. F. Hammond, president, and Dr. S. Wernick, hon. secretary, of the Institute of Metal Finishing, with Mr. M. L. Alkan (M. L. Alkan Ltd.) (Centre) Dr. L. B. Pfeil, managing director, The Mond Nickel Co. Ltd., and president elect of the Institute of Metals, with Mrs. Pfeil.

(Bottom) Mr. J. A. Barnet (left), liaison officer of the British Productivity Council, and Mr. N. D. P. Smith (I.C.I. Ltd.,)

The authors referred to the importance of cyanide concentration being maintained correctly in the solution. In practice it was even more important to keep the hydroxide concentration at its maximum value. Dr. Layton said that he had found that if the deviation from the optimum value was more than 5 per cent. such good results were not obtained.

Unless the anodic and cathodic figures were almost equal there was no advantage in using P.R. instead of d.c. from the levelling point of view. In other words, the advantage of P.R. lay not in getting smooth, level deposits for the normal thickness in which platers were interested, but only in applications such as electroforming.

He thought that the authors' explanation of the effect of P.R. in cyanide solutions and its lack of effect in sulphate solutions was further supported by Fig. 7, which showed the influence of stirring on the rate of roughening. Stirring reduced the concentration polarization, and that was further evidence which proved beyond doubt the correctness of this explanation.

In Fig. 6 he would have expected curves 1, 2 and 3 to be parallel, but they did not appear to be so. Taking specimens 2 and 3, at 75-80 μ in. they did not appear to be smoothing so rapidly as the one which started off at 75 μ in. Was there any reason for this, or was it part of the limitations of the experimental method—in other words, the use of the Talysurf instrument?

On page 15 the authors described the effect of interrupted d.c., and it might seem as if greater roughness was obtained with interrupted d.c. than with d.c. alone, but he felt that this was not true. Was there some explanation of this?

Mr. U. R. Marx (Wilmot-Breedon, Ltd., Coventry) said that the influence of *p*H changes in the boundary zone, was very critical. In an ordinary nickel bath acid had to be added from time to time and he was very interested in this effect, For example, instead of plating at a load of about 1 amp. per gal., he increased this to about 5 amp. per gal. for work not essentially dissimilar to that described by the authors. It was found that in no case over a period of 45 min. plating did a *p*H rise of less than 0·2 occur, and very often it went as high as 0·5.

In the specimens which the authors used, there were fairly deep grooves, compared with the grooves with which the ordinary plater had to deal. He wondered, therefore, whether or not the agitation which the authors provided was adequate enough to overcome the effects of the build-up of basic metal or the depletion of hydrogen ions or whatever might take place in the crucial boundary layer?

Mr. D. E. Weimer (M. L. Alkan Ltd., Greenford) said that while the experimental method used by Watson and Edwards was very suitable for differences in levelling power between different solutions, he would like to have their opinion on whether or not a commercial levelling solution needed to have a very high levelling power on their scale. For instance, on a steel surface with an r.m.s. value of 30 μ in., which had a valley-to-peak distance of 0·001 in., and the width of these scratches three to four times the height, then if a deposit of 0·0001 in. was made on this surface (ten times the peak-to-valley height) from a solution of, say, 0·4 or 0·5 r.m.s., he would have thought that adequate levelling of the base metal would occur.

As the authors had pointed out, their experimental solutions of the highest levelling power did not always produce deposits of good quality, so that it was obvious that levelling power had to be considered in relation to other properties which the solution might give to a deposit.

With regard to the authors' conclusions, he wondered whether or not they were justified in concluding that substances which, when added to an electroplating solution, were incorporated in the deposit giving an increased cathode polarization, should all behave as levelling agents. Had the authors investigated other materials in nickel solutions since doing the work reported in the paper, and had they investigated cyanide solutions?

Effect of Breakdown Products

The authors indicated that their experimental work had been carried out with freshly prepared solutions. What happened in solutions which had built up, in the course of electrolysis, breakdown products? These would have some effect on the cathode polarization, particularly since they were often present in a higher concentration than the addition agent itself. Where were these breakdown products formed—at the anode, the cathode, or both? If they were formed, at least in part, at the cathode, was the mechanism of the cathode as simple as had been postulated, that is, the direct adsorption of the addition agent? Since solutions which contained breakdown products gave inferior deposits, some adsorption of these materials seemed likely. Had the authors any experimental data on the levelling characteristics of electrolyte solutions in comparison with freshly prepared ones with the same concentration of addition agent?

Dr. S. Wernick (Consultant, London) referring to the paper by Watson and Edwards said that he was very intrigued by the photomicrographs in Plate 2, and in particular by A and B, where, using the admirable technique employed by the authors, it was easy to see where the levelling agent had entered the deposit. The authors said that the levelling agent was obviously much more concentrated in the peaks than in the recesses, but

from the pictures it appeared as though the levelling agent were exclusively concentrated on the peaks. Was this merely an optical effect or had the authors any evidence to indicate that perhaps there was a tailing off from the recess up to the peak. If in fact the photomicrographs were correct a field for speculation was opened up. He wondered whether a mechanism such as the selective appearance of the levelling agent in the peak, resulting in a degree of inhibition, so far as local deposition was concerned, at a certain stage, which would in turn result in a heavier build up of the recess, did or did not follow the theory which the authors had put forward.

Use of Radioactive Tracers

Had there been any attempt to use a tracer element technique to determine where in fact the levelling agents were mostly concentrated? He imagined that a technique of that kind might perhaps be as revealing as the very beautiful technique which the authors had described.

Referring to the paper by Hickling and Rothbaum, he was hoping that as a result of their researches it would be possible to leave the meeting feeling that it would be possible to obtain smooth copper deposits in cyanide solutions. As Dr. Hickling had indicated, it was a major problem for the electroplater, and were there in fact degrees of types of roughness? The type of roughness that the average practical plater encountered occurred periodically, almost without warning, and took the form of a sort of local nodular growth rather than a series of humps on the copper deposit surface.

Looking at the authors' curves, he expected to find now and again a very sudden jump somewhere, which would correspond to the sort of difficulty encountered in plating practice. The technique of P.R. plating had been in use some time. Many had attempted to ameliorate rough conditions by the use of P.R., and, speaking personally, thus far he had found that there had not been any very marked improvement which would make it desirable in most instances to adopt P.R. in practical plating practice.

Mr. B. E. Bunce (Gillette Industries Ltd., Isleworth) asked Dr. Watson and Dr. Edwards whether they had any comments to make on any connexions between levelling agents from the structural point of view, and particularly organic groupings, such as for example the presence of double bonds.

Dr. P. Hersch (Development and Research Department, Mond Nickel Co. Ltd., Birmingham), referring to the paper by Watson and Edwards, said that on page 22 the authors stated that the fact that coumarin was present in the deposit was demonstrated by anodically dissolving a sample of deposit in 10 per cent. hydrochloric acid; the resulting solution smelled of coumarin and fluoresced strongly under ultraviolet light. Could this be put on a more quantitative basis and the fluorescence be used to give some idea of what amount of coumarin was actually incorporated?

It was interesting to see the cathodic deposition potential so neatly correlated to the levelling action, but why was it that an adsorbed substance lowered the deposition potential? Why did it discourage the deposition of the nickel ion and thereby lower the potential? Was it a geometrical effect? Did the organic material occupy blocks of sites on the surface and therefore make the current density which had to be applied in order to deposit the metal much higher, or were there other factors? For example, did the adsorbed material act chemically by some complexing action, for instance, on the nickel ion before it was deposited?

At the Mond Nickel Company some experiments on the action of brightening and levelling agents on the anode potential and on the dissolution potential of nickel had been carried out to see whether or not those materials that have some brightening action push the potential in one or the other direction. The outcome of this was rather negative. Brightening agents were sorted out into those which raised the deposition potential and those which lowered it, and a histogram which was made showed that the distribution was rather even and that on the whole there was no net effect. It would be extremely useful if more extensive work were done on the cathode potential and levelling action on materials which were not so conventional as coumarin, saccharin and thiourea.

Mr. J. M. Sprague (Consulting Electrochemist, London) said that the action of levellers and that of brighteners was quite distinct; but in prac ice could the effects be fully separated? He would have thought that in the usual so-called bright plating solution there were both levellers and brighteners, and that the characteristics and effects of the brighteners would have some action on the way in which the levellers would work.

Mr. C. E. Moore (Ashton and Moore Ltd., Birmingham) said that in the paper by Watson and Edwards, reference was made to the fact that when cadmium sulphate was used, although considerable levelling was obtained, it varied widely across the cathode and the results were not reproducible. The authors did not enlarge on that at all, but could they give any indication of why they thought that that was so with cadmium sulphate and not, presumably, with the other levellers employed?

Referring to the photomicrographs, Mr. Moore also noted the extraordinarily sharp definition between the portion of the deposited metal in which the leveller was presumed to be incorporated and that in which it was absent. It might at first be imagined that it was purely an etching effect, but he wondered whether there was some effect

whereby once a leveller had been adsorbed on a metallic surface during deposition, it did not favour subsequent deposition of the leveller at the same points. Might that be the reason why the normal diffusion gradient which would be expected in a V-shape was not present.

MR. T. E. SUCH (Ionic Plating Co. Ltd., Birmingham) said he wished to comment on the peak obtained in the curve for levelling against concentration of added chemical in Fig. 1 of the paper by Watson and Edwards. He had done some work with a nickel solution, using coumarin as leveller and steel plates polished to the usual standard for decorative finish as a test-piece. The roughness was measured by a Talysurf before and after plating. Current densities of 4 amp. per sq. dm. were used and it was observed that a limiting value for the levelling obtainable on any surface was obtained at a certain concentration of coumarin, but he did not notice that any increased additions of that chemical produced a falling off in levelling.

Maximum levelling was obtained with different concentrations of coumarin according to the initial roughness of the surface being plated. For a surface of 30-40 μ in. 0,0007 molar of coumarin was sufficient, while 0.001 molar was needed for a 5-10 initial μ in. surface. The values given in Table IV of the paper were specific only to the surface under investigation. While the principle involved was the same, he thought the authors would agree that the figures given in Table IV would not necessarily apply to a normal polished surface being plated in the usual type of plating bath, although they did say that diamond-ruled polished copper surfaces gave the same results as the gramophone matrices. The theoretical curve in Fig. 11 seemed to agree better with normal plating conditions than did the results obtained with gramophone matrices.

Mr. Such said that with a proprietary copper pyrophosphate solution, he observed that increased additions of leveller reduced the levelling obtained, and if enough leveller were added there was no levelling at all. The best results were obtained from this solution using half the recommended concentration of leveller, which was a very surprising occurrence!

The PRESIDENT referring to Fig. 11 in the paper by Hickling and Rothbaum, in which a remarkable difference was shown in the rate of roughening of copper which had been either electropolished or annealed before plating, said that there was one condition common to both. The copper as rolled would have a Beilby film on it. When it was electropolished, the Beilby film would be removed. Similarly, if it was annealed at higher temperatures the Beilby film would be diffused into the basic copper. In either case one would finish with a relatively coarse-grained structure. If, therefore, the plating was being done in copper-sulphate solution instead of cyanide solution, it would be expected that the conditions would be ripe for grain growth and the production of large crystals, and in consequence a greater increase in the rate of roughening.

Authors' Replies

DR. J. EDWARDS (B.N.F.M.R.A., London), said in reply to Dr. Hoar, that he had no explanation to put forward for the dual action of thiourea. They were giving very serious thought to this, because obviously it might give a clue to the explanation of the whole effect and were carrying out experimental work designed to elucidate this point. He mentioned in view of Mr. Marx's comments on *p*H changes in the boundary layer, that a possible explanation, which they had in mind was the variation which occurs in *p*H over the surface in levelling conditions.

Of interest, in considering possible explanations of this effect, was the similar effect observed in the inhibition of acid attack on steels, particularly because it then applies to an anodic action as well as to a cathodic action, and this would presumably exclude some possible mechanisms.

The position of the maximum, said Dr. Edwards, if it does really exist in practice, is of considerable practical importance. The maximum is no doubt affected by other factors, but, so far as his own experience went, it is not affected by size, because the only experiments which they had done, apart from the work on the gramophone record masters, was on the diamond groove surfaces. As is stated in their paper, the maximum levelling action was found in 0·00022 and 0·0004 molar solutions, which corresponds closely with the maximum range of levelling on the standard surface.

Referring to Mr. Such's comments on this point, they were gratified that their theoretical curve was better than their practical one in predicting what was going to happen under industrial conditions, but he would have to look a little further into the experimental side of this before commenting on the reasons for the difference. It was important to find out whether or not it is true that levelling power does not decrease beyond the limiting value except in the case of the one proprietary substance mentioned in the paper. Still referring to Mr. Such's comments on that point, they would not, of course, expect their actual data to apply to other surfaces, partly because the very definition of levelling power is bound up with the shape and dimensions of the surface. It is possible to give it quantitive expression only when the precise surface in question is known. This, however, should not exclude the possibility of comparing the concentration at which maximum levelling takes place, and it should be possible to do that on surfaces of different form and size.

On the question of modifying the maximum by mixing various addition agents, something had been done on these lines, but the results had not yet been sorted out. The matter was not a simple one; the effects are not additive and it is not possible to superimpose the maxima and make a plateau. There was a tendency, if additions are made to good levellers, as is often done, to improve other properties, to destroy or reduce greatly the levelling action.

Dr. Hoar had made a remark about cadmium appearing black in microsection. They had not shown by any direct means that it was cadmium sulphide. The appearance was not entirely black, and there were cracks in the deposit which may have contributed to the black appearance. There was no yellowness which could be distinguished, but as the substance was very finely divided it seemed reasonable that it should have a black appearance.

Dr. Hoar had also asked about other metals. The question of other metal addition agents had not been investigated. In particular he had mentioned cobalt-nickel, which the authors had not examined, but Dr. Edwards said that he was not at all sure that it would be expected in practice to demonstrate very high levelling power. In any case he thought it possible that the mechanism which had been proposed might still apply, in a somewhat modified form, even when there is a really large quantity of a second metal ion present.

Mr. Weimer had asked whether or not a commercial solution need have very high levelling power on the scale which the authors had adopted. Normal commercial surfaces were only very slightly rough by the standard of the surfaces with which the paper had dealt. In a section it is only just possible to see the undulations in the surface, and when 0·001 in. of metal is plated on it the undulations are invisible in section, but not necessarily in looking at the surface. It frequently happened that pits and nodules could only be revealed in a sectioned deposit by the use of an interference technique because they were so shallow, although they would

CONFERENCE CAMEOS

(Top) Mr. A. E. Quenzer (Prodorite Ltd.) (left) and Mr. R. W. W. Sanderson (General Electric Co. Ltd.).

(Centre) Mr. C. D. S. Bridgett, of Maintenance Technique Development Establishment, in discussion with Mr. E. S. Spencer-Timms, Armament Research and Development Establishment (right) who was responsible for the organization of the technical exhibition.

(Bottom) Mr. A. H. Lupson (Electrochemical Engineering Co. Ltd.) (extreme right), chairman of the Conference Social Organizing Sub-committee, with Mr. H. Cann (W. Canning and Co. Ltd.), a member of the sub-committee, and Mr. H. Silman (Electro hemical Engineering Co. Ltd.) and Mr. R. A. F. Hammond, pre.ident I.M.F.

be clearly visible to the naked eye. That meant that a levelling action, and perhaps a very strong one, was still required, although it would be operating on a much shallower scale.

So far as addition agents were concerned, up to now more than 50 in nickel solutions had been examined, and in all cases where the addition agent lowers cathode potential or increases polarization and is co-deposited in the coating, levelling action will occur. A little work had also been done on copper plating from a sulphate bath, and again the predictions had been verified in the few cases which had been studied, although so far no cyanide solution had been investigated. It was true that levelling must be looked at in relation to other properties, and most of the deposits which had been produced would not have been any use to anyone, one thing had to be taken at a time. More and more attention was now being given to the examination of other properties of the deposits.

All the work had been done on new solutions, but in other work the effect of the decomposition of addition agents and so on on the properties of the deposit had been fairly closely studied, but that was really a separate subject.

Dr. Wernick and Mr. Moore had commented on the illustrations in the paper showing the distribution of incorporated matter in the deposits, particularly B in Plate II, with quinoline ethiodide, where a very sharp boundary is apparent between the black and the white areas. The probable reason for this is that when quinoline ethiodide is added it first blackens and then is decolourized, so that on the surface of the photomicrograph there is a dynamic effect of the production of the black material, whatever it is, and then its dissolution. Where there is a good deal of material it is being produced more rapidly than it is dissolved, for a time at least, so that there is time to build up a fairly thick deposit on the top of the surface. On the peaks, where there is less of it, this is not so, and only a little is produced before it is dissolved. The tendency of these two opposing processes is to produce a sharp boundary. That in itself, perhaps, does not give very good evidence for this uniform distribution, though it explains away this particular piece of evidence.

On the other hand, the microsections taken through deposits containing thiourea are much more convincing. There is a dark appearance on the peaks which spreads with etching time down to the recesses, and, what is more important, there are lines, spectral bands of colours, as one passes down to the recess, and with increased etching time these move down. Radioactive tracers had not been used. It would be interesting to show this effect in that way, but there was concern whether this technique could be applied on a micro scale.

Dr. A. HICKLING (Liverpool University), replying to the discussion on the paper by Dr. H. P. Rothbaum and himself, said that Dr. Layton had commented on the fact that the process did not normally involve electrolytic polishing. That was now generally accepted, but it was worth noting that, as mentioned in the paper, under extreme conditions—conditions which are not normally encountered and which are difficult to obtain—it is possible to make a specimen enter the polishing region. This was not likely to have much practical application, because the conditions are too difficult to maintain readily.

His remark about cyanide and particularly about hydroxide concentration was an important one, with which the authors fully agreed, and it was partly responsible for the technique which had been employed of using small quantities of fresh solution in every experiment.

What Dr. Layton said about stirring, showing that it is a concentration polarization effect, was in line with the authors' conclusion, that once the initial stage was over the roughness was largely due to concentration polarization.

Dr. Wernick had referred to different types of roughness. All of them were extremely important and many of them should in fact be investigated, but the authors had chosen the one which they thought would be easiest to investigate. Nodular growth was inevitable and it often showed up in the Talysurf records by the needle showing suddenly an enormous peak.

Dr. Wernick had asked why P.R. is not more widely used in practice. From the point of view of a practical electroplater, the answer was probably that if all that was required was to get a thin metallic coating which was smooth and bright, this could be achieved far more easily by other methods. Where P.R. was important was in electroforming, where it is required to build up a considerable thickness and to restrain the development of roughness over a considerable period by electrolysis.

Abstract No. 3:

Production and Properties of Opaque Coatings by Chromic Acid Anodizing

By A. W. Brace, A.I.M., C.G.I.A. and R. Peek**

IT has been reported that attractive coatings with good response to dyeing could be obtained using a 10 per cent. CrO_3 electrolyte at 130° F (54° C) and a constant voltage of 30 volts. However, as little information was available on the film

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thickness obtained, response to various alloys, control of bath of properties of the coatings produced, etc., experiments were carried out to evaluate the commercial possibilities of the process. Such factors as the effect of anodizing temperature and voltage, influence of Cr_2O_3 concentration, response of various alloys, flexibility, deep-drawing properties, abrasion resistance, heat resistance, light fastness, etc., were investigated.

The results of the experiments confirmed the claim that attractive opaque anodic coatings can be produced in the above type of Cr_2O_3 electrolyte. Under the conditions previously mentioned a coating 5μ thick can be obtained in 20 min. and a coating 10μ thick in 60 min. The coatings produced are flexible, heat resistant, and can be dyed satisfactorily. They also have good atmospheric corrosion resistance, although they are too thin for really long-term exposure.

The fact that the film produced can be so readily dyed, compared with the normal Bengough film, is probably due to the fact that the pore volume is about twice that of the Bengough coating, which will appreciably increase its ability to absorb dye. In addition most of the Bengough coatings are only 3 to 5μ thick whereas these coatings are 5 to 10μ thick. This increased thickness also increases the density of colour.

The opacity of this film is also of interest since there appears to be no reason why the coating, which virtually is pure aluminium oxide, should be opaque. Detaching the film by shattering it in an electropolishing solution shows that it is transparent but the surface of the metal is etched. This fact is also confirmed by chemical stripping of the oxide film in the chrome-phosphoric acid mixture. The etching of the metal surface does not take place in the first 5 min. of anodizing because the coating is then transparent and the reason for the subsequent development of the opaque appearance is at present being investigated further.

There is no doubt that there are attractive possibilities for the application of this process for the production of a new range of colour-anodized finishes for hollow-ware, jewellery, refrigerator parts, automobile trim, etc., using up to 20 to 30 min. anodizing treatment. The flexibility of the coating could also be exploited for applications where some forming is required on the finished article. Most aluminium alloys can be treated satisfactorily, but the manganese in the alloy NS3 tends to produce a greyish coating whose depth depends upon its metallurgical history and the amount of impurities present. Gravity die castings respond very well provided they are free from porosity.

Discussion

Mr. V. F. Henley (W. Canning and Co. Ltd.) said that he assumed that air agitation was used, but that the curves in Fig. 1, and also in Fig. 2, indicated that perhaps the degree of agitation was not so great as would be used in commercial production. The initial rate of increase of film thickness in Fig. 1 at 140° F was somewhat less than would be expected. At that temperature the conductivity of the solution was higher than at 130° F, and one would expect the current density to be higher and therefore, at least at the beginning, the film thickness and the rate of its increase to be greater. If, however, the agitation were insufficient, the solvent effect of the electrolyte, coupled with the considerable heating effect at the interface of the film, would result in the type of curve which the authors obtained. In Fig. 2 it was also noteworthy that the curve at 40 volts and a current density of 18 amps. per sq. ft. did not quite fall in line with what he would expect.

The paper quoted figures for the consumption of chromic acid. It was interesting that after treating 125 sq. ft. of metal there was a chromic acid loss of about 1 lb. Judging by the normal rate of dragout on metal surfaces, it was probable that at least half that 1 lb. was dragged out of the solution and not consumed, as it were, by reduction of the cathode. In other words, there was a possibility of improving the economics of the process by additional care in regard to dragout or by the provision of some form of dragout trap.

Another interesting statement made by the authors was that the accumulation of aniline and chromate had no deleterious effect on the film formation or properties of the coating. That was in direct contrast to the results obtained with the 3 per cent. solution normally used in this country.

Mr. Brace, in introducing the paper, had referred to the anodizing of various alloys and suggested that by reducing the operating voltage it was possible to overcome the difficulties which were normally experienced in treating some of the copper-containing alloys. It was quite customary to reduce the temperature of the electrolyte when dealing with some of these rather difficult alloys, and at the reduced temperatures quite good coatings were obtained in a 3 per cent. solution, while the hardness of the coating was improved by operating under these conditions. Had the authors tried treating some of these alloys in the 10 per cent. chromic acid solution at lower temperatures than the average of 130° F which they proposed for normal types?

Dealing with dyeing, the authors had given a list which contained dyestuffs which were quite commonly used on sulphuric acid coatings, but in

his experience some dyestuffs were sensitive to chromic acid, and it would be realized that in commercial production there was always the possibility that traces of electrolyte would be dragged into a dye bath. Had the authors tried the effect of adding small quantities of chromic acid to these various dye solutions, to make certain that they were not adversely affected? Otherwise, some people might run into difficulty if they attempted to use the dyestuffs prescribed.

Sealing of Coatings

On the subject of sealing, the authors had chosen an unusually low concentration of nickel acetate. The normal concentration, in this country, of nickel acetate is about 0·5 per cent. at a *p*H of 5·6–5·8. The effectiveness of this solution is emphasized by the *p*H, and in addition there is an appreciable adsorption of nickel into the anodic coating, or in other words a continual loss of nickel. It is probably there in the form of nickel hydroxide. If a 0·1 per cent. solution were used it might well be somewhat unstable when used on a production scale, and constant control would have to be exercised to maintain it at its initial concentration.

In connexion with the question of sealing sulphuric acid coatings, the sealing solution in fact decided on is potassium dichromate. Hitherto potassium dichromate has not normally been specified for the sealing of chromic acid coatings, whereas, of course, it is common for sulphuric acid coatings. The contention in the past has been that the corrosion resistance of chromic acid coatings is all that could be desired. However, anodic coatings are now required to withstand conditions more arduous than hitherto, and additional corrosion resistance might be desirable. Had the authors considered the use of potassium dichromate sealing with their anodic coatings, to increase their already substantial corrosion resistance?

The authors had mentioned the specific weight increase of the coating due to the adsorption of water by the anodic film. It was interesting to note that the weight increases obtained with that particular form of chromic acid coating were approximately double those with the sulphuric acid coating. Was that due to the difference in pore size of the coating mentioned in the paper, or was there some other explanation?

At the end of the paper, and liable to be overlooked, was a most important observation, that the "opaque" chromic acid film is not opaque at all, but merely looks opaque. The authors had separated the film from the basis metal and said that it is transparent, and that the opacity is an optical effect arising from the etching which has occurred on the basis metal during the anodizing process.

If sulphate is added to chromic acid anodizing solution the apparent opacity of the film decreases and it becomes transparent. Had the authors tried removing all traces of sulphate from their solution by the addition of small quantities of barium hydroxide to see if that would increase the apparent opacity?

Mr. J. M. SPRAGUE (Consulting Electrochemist, London) said that sealing in steam was sometimes recommended to avoid the inevitable change in *p*H which takes place rapidly in boiling water. Had the authors done anything on that, and had they special reasons for using the hot water method?

Dr. S. WERNICK (Consultant, London) said that the remarkable difference between the curve for 140° F and those for 120 and 130° F in Fig. 1 was interesting. Why a matter of 10° F should result in the rate of growth being sharply reduced was something on which the authors might care to comment.

Deterioration of Bath

In the results reported on the 50-gal. bath, the important thing was that there should be an age effect, so that it might be possible to know what deterioration was likely to occur as the bath was used. The bath in question had dealt with 90 sq. ft. of material in 50 gal. of solution, which worked out at just under 2 sq. ft. per gal., which would be a very small amount compared with practical conditions. It was therefore important that the authors should continue that work and determine whether at very much larger rates of use, where the areas are much larger for a given volume, there was no deterioration in such things as the rate of film formation. The effect of aluminium salts in particular had to be watched to see how far they affected both the quality of the film and the rate of film formation.

The authors had stated that dyeing black needed a dyeing time of about 15 minutes at a temperature of about 150° F. Had they tried using a much higher temperature, which is normal practice in producing a black dye on sulphuric acid and, indeed, chromic acid type films?

Mr. W. N. BRADSHAW asked what was the amount of metal which was taken into solution during the process, and what proportion the metal dissolved into solution bore to that which formed the film. In other words, what was the film-forming efficiency?

Mr. M. G. ASHTON (Ashton and Moore Ltd., Birmingham) asked whether, in view of the thickness and porosity of the film, any trouble had been encountered with chromic acid adsorption in the film.

Also had any work been done on the impregnating of that film, or films of that sort, with phosphates or oxalates to ease fabrication?

Authors' Reply

Mr. A. W. BRACE, replying to the discussion, said that there were two reasons for the peculiar behaviour in relation to the rate of film growth at higher temperatures and higher forming voltages. The first was that the curve of the rate of film growth of any anodic coating has this general characteristic tended to flatten off with time.

The period up to which that occurred was compressed or extended according to the anodizing conditions, primarily the strength of the electrolyte and the temperature of the electrolyte. In other words, there was a point where the rate of film formation due to the outward flow of current was being balanced by the tendency of the electrolyte to re-dissolve that film.

The second factor which came in was that reactions at the cathode were probably as important as reactions at the anode. Anodizers tend to study what happens at the anode; but other work had tended to show that the reduction of the chromic acid at the cathode resulted in a lower efficiency, due to a resistance imparted, probably, by cathodic polarization, which meant that although more current might be consumed it did not necessarily result in faster growth of film.

The question had been raised on the accumulation of aluminium. Checks on the response to dyeing, the film thickness, and a limited number of corrosion tests, both accelerated and out of doors, had so far revealed no appreciable difference. The bath had been running for 21 months or more, and putting 90 sq. ft. through it and measuring how much chromate was lost had been merely to put a lot of work through in quick succession. The number of square feet which had gone through that chromic bath was in the region of four figures, and it had reached a point where it contained something like 10 gm. per litre or more of aluminium and it still performed very effectively.

On the question of alloys, the effect of lowering the electrolyte temperature in order to deal with the more difficult alloys had not been examined. There was a tendency on the part of commercial anodizers to fix a process at so many volts, but in the authors' opinion it was decidedly better and more helpful to fit the current density and then adjust the voltage to give a current density which gave the right rate of film growth for a particular alloy.

Both Mr. Henley and Dr. Wernick had referred to dyestuffs. Mr. Brace strongly supported what Mr. Henley had said about chromic acid sensitivity. There are certain dyes not included in the list in the paper, which do show sensitivity to chromic acid, and that had been shown in his experience in anodizing small articles, in which small amounts of chromic could be trapped. Certain dyes worked very well with chromic and others were definitely unsatisfactory.

With reference to the black dye, he strongly disagreed with the tendency in some commercial practice to use a very hot electrolyte to get a porous film to produce a good black, because, while it was possible to do that, his experience was that from the points of view of consistency of colour, freedom from a tendency to get a yellow or blue tinge, and ultimate corrosion resistance and ease of subsequently sealing the coating and preventing bleeding out of the dye, a coating produced under conditions which give a good, sound structure was much more satisfactory than one where there was excessive porosity.

On the question of sealing, and in what he said about nickel acetate Mr. Henley had been correct in his criticism. Use of the 0·1 per cent. nickel acetate was justified in the laboratory, because it had been found that there is a greater tendency for a slight chalkiness to appear on the surface if nickel acetate sealing is used, and it increases with the nickel acetate concentration. He would normally recommend 0·5 per cent. nickel acetate, suitably buffered to give the correct *p*H.

The interesting point about the dichromate was worth further investigation, because he was convinced that there was something in the sealing of anodic coatings which was important, and it was not solely a question of porosity. The Bengough film has 3–4 per cent. pore volume as against 7 per cent. for the 10 per cent. chromic film, but what had been found was that there was some chemical change which took place in the anodic coating on sealing which appreciably enhanced its corrosion resistance. In a SO_2 humidity test the unsealed coating blued badly but the sealed coating was unaffected. The same effects were found with sulphuric coatings and, though there is less porosity in the Bengough film, there would be an improvement with dichromate solution, hot-water sealed.

Sealing in Steam

Mr. Sprague had asked about sealing in steam, as against sealing in water. The reason for advocating steam sealing was that if the *p*H of the sealing bath got below 5, much of the beneficial effect of sealing was lost, just as happened if the sealing temperature dropped to 80–90° C. The disadvantage found with steam, however, was that the rate of sealing depended very much on the arrangements made for steam sealing. Whereas with an ordinary anodic coating it was possible to have quite a fast rate of sealing, with steam sealing, depending on how efficiently the tank in which the sealing was done was closed, it could be a very much slower process before reaching the steady point, which should be the point to aim for to get good sealing.

Mr. Bradshaw had raised the question of the coating ratio with this process. While no really careful checks had been carried out it could be assumed that in the linear portion up to 20 min. the anodizing efficiency was not very different from that of ordinary sulphuric-acid anodizing, and that again meant that in terms of film thickness there was not a great deal that was lost.

With regard to the question of adsorption of Cr_2O_3 in the coating itself, Mr. Brace said that that film is not in fact more porous than the sulphuric film. A 20 per cent. (by volume) sulphuric acid electrolyte, at 12 volts gave about 13 per cent. pore volume, whereas for that coating the figure was 7–8 per cent. The difference was that the Cr_2O_3 coating had a pore of about 248 Å diameter, as against 120Å for a sulphuric-acid coating.

The coating was virtually pure aluminium oxide and one or two checks had failed to reveal any appreciable traces of chromic acid in the film. It was certainly below 1 per cent. of the film weight, whereas a sulphuric acid coating had 13–16 per cent. of SO_3 estimated chemically as such, which in terms of aluminium sulphate must be of the order of 25 per cent. It was because the aluminium oxide film was of such purity that it had such high corrosion resistance. Impregnation with phosphate or oxalate had not been attempted and he doubted whether the technique of using that film and impregnating with phosphate would deal with the problem which Mr. Ashton had raised; a straight chemical conversion coating on the aluminium would probably be a more successful approach to his difficulty.

Dr. Wernick had commended the wisdom of restricting the use of that coating to interior applications, and had emphasized the merits of the Bengough film. It was surely not reasonable to compare the corrosion resistance of a 5-μ Bengough film with that of a 20–25-μ anodic film used widely in the U.S.A. for the protection of permanent buildings, particularly for colour anodizing; and a covering which had withstood 12 months at Stratford, in East London, without showing any deterioration might be considered better than the general run of chromium plating on the outside of motor cars!

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Abstract No. 4:

Metal Coatings Produced From Powder By Peening

By G. H. Jenner, B.Sc., and T. P. Hoar†, M.A., Ph.D., F.R.I.C., F.I.M.*

PEEN plating is a new method of plating hardware with such metals as zinc and brass without the use of heat or electricity. The process results from the combination of the well-known technique of barrel finishing with the principle of cold welding. Plating is performed in a modified hexagonal tumbling barrel in which much larger loadings are undertaken than with normal tumbling operations.

The barrel is rubber lined and has a variable drive since different products require various speeds of rotation to give satisfactory results. The metal which is to be plated on to the articles in the barrel is in the form of powder, preferably produced by condensation of the distilled metal or by atomization of the molten metal. It is necessary for the particles to be spherical or pear-shaped, and in addition, the metal particles should be as free from oxide as possible. Like the more modern barrel-finishing techniques the process is carried out under water, and chemicals, called "promoters", are added to the water to facilitate the cold welding of the powder to the basis metal.

Steel particles, only, are referred to in the paper, and the pre-plating procedure recommended consists of degreasing, acid pickling followed by an immersion copper coating, which aids the "strike" of a plating metal such as zinc. Also added to the barrel load is an impacting material such as crushed steel, grit, aluminium oxide chips, glass balls, etc. Metals which may be used for peen-plated coatings include zinc, brass, cadmium, tin and lead: aluminium may also be plated by a modification of the method. The paper gives full details of typical barrel loads for these metals.

A peen-plated coating is, in general, suitable for any of the after treatments usually given to electroplated articles.

Peen-plated coatings are built up by the successive welding of small pieces of metal on to the object, and this leads to a characteristic laminated structure. Particles of metal may become welded together without being deposited on the object, and, depositing at a later stage, give rise to a difference in "grain" size within the coating. Heat treatment at 428° C for a few seconds drastically increases the apparent "grain" size.

Good uniformity of coatings is obtained on objects of irregular shape and adhesion of the coatings is of a very high order, and for equivalent thicknesses of metallic coatings, the peen-plated coatings have, in general, a superior corrosion resistance.

Peen plating has some similarity to metal spraying, but in the latter process the particles of metal employed are larger and thus a rough surface is required to obtain satisfactory adhesion. It is probable that a peen-plated coating could be

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† *Cambridge University.*

secured on a perfectly flat surface provided that this surface was perfectly clean. In practice a sufficiently clean surface is only obtainable by some treatment such as acid pickling, and it is often advisable to follow this with the deposition of a thin film of copper to limit oxide formation on the steel. The soft oxide of copper is easily removable during the process and copper is also more susceptible to cold welding than is the steel base to be plated.

Another aid to plating is the inclusion in the barrel of a small amount of very soft metal, for example, lead or tin, but even with these aids no coating is formed without the addition of "promoters". These are mild pickling agents with added inhibitors which remove the oxide film on the basis metal while giving little or no continuing attack on the metal itself.

To be suitable for peen plating the basis metal must be easily pickled and the particles of the metal being plated must be soft enough to be deformed by the tumbling operation. Also the shape and size of the article being treated must be such that it permits fairly easy access of the impacting material. One of the main advantages of peen plating is the resultant freedom from hydrogen embrittlement of the coating. Furthermore, sintered parts may be plated with ease as also can carburized and nitrided objects. The ability to deposit unorthodox combinations of metals is expected to become of significance in industry and also under special conditions the process can be used for deposition of metals and alloys on to glass, ceramics, etc.

Since peen plating is essentially comparable, from a plant point of view, with barrel finishing, it is intrinsically cheap and simple, and in the U.S.A. cost savings of up to 60 per cent. have been reported compared with those methods used previously.

Discussion

Mr. E. A. OLLARD (Atlas Plating Works, Ltd.) said that the theoretical ramifications of the subject were of very great interest, and he hoped that the authors would in due course enlarge on them because the whole mechanism which causes metal to adhere, as it undoubtedly does in these circumstances, should throw a great deal of light on other problems.

The process, Mr. Ollard said, undoubtedly produces quite a satisfactory coating, on a par with zinc plating or galvanizing, or brass plating in the case of brass, so that the main interest was an economic one. He had come to the conclusion that the economics of the process depended on the amount it was necessary to plate by this method. He did not believe that peen plating had any advantage over ordinary barrel plating when dealing with up to one ton a day, but when dealing with several tons (say 5 tons) a day it had a very definite economic advantage.

If the cost of peen plating was compared with the cost of electroplating in a barrel, it would be found that the cost of preparation was about the same. The time was also not very different, being about 45 minutes, which was the average for ordinary commercial plating. The cost of the zinc would be about the same, because it was no cheaper in powder form than as an anode.

Mr. R. E. MANSFORD said that the authors had mentioned similarities between their process and metal spraying. A very similar process was carried out with metal spraying in a barrel before the war; in this country it was used as recently as 1947, and it was still being carried on in South Africa. The main difference was in the method of obtaining adhesion. The great advantage of the authors' method was that it was not necessary to grit blast to obtain adhesion, the usual practice in metal spraying; but against that it was possible to mention substances such as stainless steel and titanium, where the oxide film was troublesome with the peening method.

Mr. G. H. JENNER said that the paper was mainly concerned with a process done under water. He asked Mr. Mansford if any heat was involved in his process.

Mr. MANSFORD, continuing, said the paper had stated that "The particles of metal employed in metal spraying are considerably larger than in peen

Selection of articles coated by the peen-plating process on show at the technical exhibition staged in conjunction with the conference. Samples include zinc-coated clout nails and lengths of brass-coated chain. The types of powder suitable for use with the process were also on show.

plating." He questioned that, because the powders used could be produced by atomization of molten metal, which was the same process carried on in metal spraying. In metal spraying from the wire process the particle size was about 20 microns, and from the photomicrographs in the paper he doubted whether the particles were very much smaller than that. The authors said that the particles in metal spraying must be heated, but that was not necessary. They were heated merely to melt them from the wire or propel them through the gun, but when they reached the job they were comparatively cold, and it was possible to spray one's fingernails with zinc and to spray fabrics.

Had the authors found that the adhesion with zinc was better than with some of the other metals which were mentioned? With metal spraying there was better adhesion with zinc than with other metals, where the method of surface preparation tended to be awkward. As regards the thickness of the coating did the adhesion fall off as the thickness increased, perhaps through the build-up of stress in the deposit?

Dr. S. WERNICK (Consultant, London) remarked that the authors had said that very fine powders were the most suitable for the operation of the process. What mesh would they normally employ? The authors also referred to "the deposition efficiency of the plating metal", and he did not understand what method they used to calculate the efficiency of the process on a basis which had presumably nothing to do with deposition at all. As regards the white froth produced, of what did it consist? Was gassing involved, and, if so, was it at all affected by the *p*H of the aqueous phase of the mixture used?

From the paper the highest contributory factor to production costs appeared to be the promoter, and he wondered what the promoter was.

Finally, Dr. Wernick said it would be useful to have some yardstick by which to judge costs, and it would be an advantage to have comparative figures, say of peen plating compared with hot galvanizing and also with electro-galvanizing.

Fatigue Life of Springs

Mr. R. G. SLINGSBY (Geo. Salter and Co. Ltd., West Bromwich) said that since the process included the term "peening" it must be of interest to spring manufacturers. A spring was one of the most highly stressed pieces of engineering equipment, and if a spring was subjected to a shot-peening process its fatigue life was increased. He would like to know whether or not the authors had done any work on evaluating the intensity of the compressional stress produced by the peen-plating process. If not, could he suggest the use of test strip, similar to the test strips used to evaluate the stress produced in electrodeposits.

If the intensity of stress produced by the peen-plating process was good enough it was obviously of value to the spring maker, because he could protect his spring and increase its fatigue life at the same time. The authors mentioned that a spring was subjected to 10^6 switch operations. That did not mean a great deal, because it did not indicate what the maximum stress of that spring was or the stress range under which it was operating. His company had plated millions of springs under the correct conditions and they had done ten million operations, and probably more, without fracture, so that unless the peen-plated springs were directly compared with springs in the unplated condition and in the electroplated condition he did not see much value in this statement.

He drew attention to the fact that the reduction in life of springs due to hydrogen embrittlement was not entirely due to the plating process, but often due to the preliminary operations. The work of Jackson and others who followed him had shown that cleaning, pickling, etc., had as much influence, if not more, on embrittling the springs as the actual plating operation. Unless these effects were watched, therefore, the idea might be given that if a spring was peen plated it would be entirely free from hydrogen embrittlement, but that might not be so.

Dr. D. N. LAYTON (Ionic Plating Co. Ltd., Birmingham), referring to impacting material, said that the authors stated that the impacting material built up the coating of zinc or other metal and then did not build up any more. Could the authors explain this?

He did not entirely agree with Mr. Ollard's economic analysis. The authors stated that the promoter presented no effluent problem, and he was sure that platers would be pleased to hear that, but they would not be so pleased to hear that 5 per cent. of the zinc was thrown away. What were the dangers of explosion in the barrel, due to the presence of finely divided zinc powder and water?

The authors mentioned that aluminium could be deposited, but they seemed very cautious about it. Could they enlarge on this? It would be of interest to metal finishers, because aluminium was not easily and economically deposited by electrolytic means. Finally, could any information be given on the hardness and brittleness of these deposits? He thought that peen-plating was a slower process than electroplating.

The CHAIRMAN (Mr. A. W. Wallbank) said that the peen-plating process was developed by Eric Clayton and Ralph Pottberg, of Baltimore, the nephews of Tainton, the man who tried to persuade steelmakers in this country to adopt his process for

the continuous zinc plating of strip from the ore.

Peen plating, he thought, was an intriguing method of putting a soft metal on a hard one. It had great possibilities with alloys, and for uses on the fringes of the industry and where existing processes of plating were not altogether satisfactory. He was not convinced, however, that there was much of a place for it between barrel plating on the one hand and flake galvanizing on the other, the two prominent methods of plating zinc in bulk on steel articles.

His reasons for saying this were economic. Looking at the production cost analysis given in the paper, some of the overheads were shown, but there were many which were not. Second, with regard to material costs, with barrel plating these were the cost of zinc metal, and a pure anode was not necessary for barrel zinc plating; but peen plating needed a highly pure zinc powder or it did not work. Items 2, 3 and 4 in the authors' analysis were also material costs, and they were very high; in fact they amounted to four or five times the metal cost. It would not be possible to barrel plate if material costs, other than anodes, were four or five times the anode costs. The only cost in barrel plating to put against that was about 1s. 6d. per cwt. on work of this nature to cover the current and the dragout losses from the barrel solution. As Mr. Ollard rightly pointed out, the cleaning costs were equivalent, and the drying costs and the degree of mechanization for either process to get labour costs down would be equivalent.

He did not see, therefore, how peen plating could be competitive with straightforward barrel plating or flake galvanizing. It was necessary to reach every part of the contour of the article being peen plated, which meant that with a screw it was necessary to have a subsidiary load which the man had to handle, but which was not a pay load, to reach the interstices of the screw and the slot. With simple things such as nails, where peening was easy, how could it compare with flake galvanizing, where the nails were simply thrown into the drum and a load of flake thrown in?

Authors' Reply

Mr. G. H. JENNER, replying, said that it was not possible to say much about the precise mechanism which went on inside the barrels; it was largely an empirical process, which had been found to work and had gradually been improved.

There had been a good deal of reference to cost. Mr. Ollard had suggested that the process would not be economic except with a load substantially over 1 cwt. He would say that it became economic at between 1 and 1½ cwt. on ordinary, normal zinc plating.

The process was of great advantage with chains. There was not, as far as he knew, any substantial plating of chains in barrels, whereas with the peen-plating process it was possible to barrel-plate chains instead of using the vat process, which was much more expensive. The American plant, which was shown as an illustration, operated at about half the cost of electroplating chains, despite the price of the promoter.

On the question of powder size, there was some peen-plating powder in the exhibition, with some coatings. The intention was to get all the metal powders as fine as possible, and the zinc powder in particular had a particle size of about 5 microns which was much smaller than the powders used for metal spraying.

He could not agree that better adhesion was obtained with zinc than with other metals on metal spraying. He would draw Mr. Mansford's attention to the fact that it was possible to spray steel with very much higher adhesion than zinc, aluminium or the other softer metals. He had no information about the relative adhesions of peen-plated coatings. It was difficult to measure their adhesion, because the first problem was to plate the test-piece, and on the ability successfully to plate the test-piece depended the answer obtained from the test. It was true that increasing the thickness of the coating decreased the adhesion, and there was a limiting thickness which could be deposited by the method, for two reasons. As would be seen from the paper, all thicknesses were deposited in about the same time for the same metal. Beyond about 0·005 in. with zinc, the whole process went wrong and there was no deposition at all; there was too much metal there to impact through to the base metal and to obtain any adhesion. He would be pleased to arrange for Dr. Wernick or anyone else to see the process in operation.

Peen Plating Efficiency

To obtain the deposition efficiency the amount of zinc put into the barrel was weighed and so was the amount on the object in the end; a simple calculation then gave the amount lost, which could be expressed as a percentage, and thus the deposition efficiency. The froth was caused by the detergent.

The promoter cost shown in the example at the end of the paper was the proprietary cost. The real cost was much lower. He did not think that it was reasonable to produce comparative costs. He was a peen plater, and if he produced an electroplating cost none would agree that it was a true electroplating cost. The same was true of a galvanizing cost. He knew that this was so, because he had heard discussions on eledtroplating and galvanizing costs. He would say that it was possible to get down to 10–12s. per cwt. for 1½–2 cwt. loads, and beyond that it was possible to get slightly lower, but not much.

With regard to springs, there was some evidence available from America that after peen plating on a shot-blasted surface the fatigue life of the springs was increased. There had been no work on this completed in this country, but some work was now under way. If anyone else would like to start work on the fatigue life of springs he would be very happy to supply peen-plated samples to have this evaluated. The figure of 10^6 referred to in the case of springs was an acceptance test by the manufacturer.

Dr. Layton referred to the impacting material and the build-up of metal coating on it. This applied only to metallic material, which was hardly used today; his company mainly used glass, and there were examples in the exhibition; they did not become plated in the course of the operation. The metallic impacting material was normally very hard wire cut to short lengths, and it was much more difficult to plate on a very hard than on a softer surface. This tended to limit the amount which could be applied. Further, when using impacting material there was always some transfer from the impacting material on to the object, and for that reason a thickness which was not exceeded, was achieved.

With regard to explosions in the barrel, in the author's laboratory there were marks on the wall due to the fact that there had been a substantial build-up of pressure in the barrel, possibly owing to the fact that the surfaces were not very clean and the process was unbalanced. It was possible to get a large pressure in that way, though it was rare, because not often were objects put in which were not susceptible to peen plating. Once the zinc had deposited on the surface of the object it substantially reduced the surface of zinc in the barrel, and if there happened to be a very slight reaction between the water and the zinc there was not very much zinc surface to affect that reaction. That explained why when the zinc did not deposit on a dirty object there was a very large surface and the zinc reaction with the water was correspondingly larger.

Aluminium coatings on steel and other surfaces were not dealt with very fully in the paper because there was not as yet much information on the subject. He believed that there was a commercial installation just starting in America for aluminium on steel for a particular application, but there was no commercial plant in this country plating with aluminium. Despite the fact that there had been five or six years work on this process, it was still comparatively new.

He had no information about the hardness and brittleness of peen-plated coatings, but if Dr. Layton would care to take samples away and measure the hardness and brittleness and let him know the result, he would be very pleased.

Mr. Wallbank said many hard things about the economics of peen plating. He thought that handling, to which Dr. Layton referred, was a red herring. If a trolley was able to take the entire load, it did not matter what the load weighed. Also if there was mechanical assistance to lift it, the operator would not know whether he was dealing with ½ cwt. or 10 cwt. The savings from peen plating accrued not from the direct materials but from savings in labour and the overheads on that labour and from savings in capital equipment and so on.

Abstract No. 5:

Electrodeposition of Tin as a Bright Coating

By A. M. Harper, B.Sc., Ph.D., A. Mohan†, M.Sc., D.I.I.Sc., and S. C. Britton*, M.A., F.R.I.C., F.I.M.*

It has been claimed that tin could be deposited bright from acid electrolytes containing both a lyophilic and a lyophobic colloid, and wood tar or its extracts has been cited as a possible source of the lyophobic material. Earlier workers had obtained bright deposits from stannous sulphate baths in which the tar produced by the destructive distillation of hardwood was dispersed with the aid of octyl sulphuric acid. Indications were obtained that the agent responsible for brightening the deposits was a neutral fraction distilling in the higher boiling-point range and also that similar compounds might be formed in the heat treatment of cellulosic products other than wood.

The authors of this paper therefore carried out an investigation to find the extent of the occurrence of the brightening agent, and to seek its identity. They also investigated the operating conditions to give consistent results. Plating was carried out in laboratory and "pilot scale" plants.

Although the investigation, especially in respect of isolation and identification of the active component of the tar, is being carried further, sufficient information has been gained to lay the foundation of a practical process for the plating of bright tin. Difficulties that may arise are the preparation of the sulphated alcohol required, the selection of a suitable source of tar and the accumulation of unwanted tar products in the electrolyte over a long period of operation. It appears that the selection of a crude tar from which only water and light oils have been removed should give consistent results and that the sulphation of an alcohol may prove to be unnecessary if available commercial products fulfil their promise.

Evidence has been obtained to show that the active ingredient in the tar may be lost in some

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processes carried out on the tar, but it seems likely that it is always present in the whole tar distillate from either hardwood or softwood, or in fractions of it distilling in the range 150 to 200° C.

In order to obtain the brightening effect the tar must be dispersed in the electrolyte with the aid of an acid sulphate of a higher alcohol. A suitable product is obtained by treating octyl alcohol or a mixture of alcohols with sulphuric acid. The amount of the sulphated alcohol used must be related to the amount of tar to be introduced, but the permissible limits are sufficiently wide to permit the employment of the process without precise analytical control. Either excess or deficiency in the amount of the coating and incorrect preparation of the material may result in the production of brittle deposits.

Brittle deposits may also be produced by excessive additions of tar or by plating at current densities higher than those appropriate to the solution used. Streaking of deposits, especially at higher current densities may occur and cathode agitation is necessary to obtain the best finish. Prompt rinsing and cathodic cleaning after plating are necessary to avoid staining of the finished work and to obtain full brightness.

The addition agent has been found effective in sulphate and in chloride/fluoride plating baths. For sulphates the following composition has been found satisfactory:—

Stannous sulphate ...	100 gm. per l.
Sulphuric acid	140 gm. per l.
Wood tar	8 gm. per l.
Octyl alcohol (sulphated)	10 ml. per l.

With this electrolyte at room temperature bright deposits without brittleness are obtained over the current-density range 10 to 60 amp. per sq. ft. At the time of writing the paper experience with volumes of electrolyte above 4 litres has been limited to 5-weeks operation of a 90-litre bath, but this was entirely satisfactory. Barrel plating in the same solution at average current densities of 12 to 18 amp. per sq. ft. was also found to give satisfactory deposits.

The addition agent gradually becomes exhausted; there is a slight deterioration during idling, but most of the loss of effectiveness results from plating out and from drag-out. Replenishment may be carried out when the brightness of the deposits deteriorates and Hull Cell tests provide the most useful means of assessing the needed amount of addition.

Chloride/fluoride baths require more careful control and further experience is needed before offering firm recommendation, but on a 4-litre scale, with agitation of the cathode, bright ductile deposits have been obtained over the current-density range 20 to 100 amp. per sq. ft.

The bright tin deposits have a grain-size very much smaller than those of matt tin deposits and the crystals are strongly orientated. The hardness of the bright deposits is appreciably greater than that of matt deposits, and the porosity , thickness for thickness, is similar to that observed on matt deposits from the ordinary stannous-sulphate bath.

Although the preparation of the addition agent and the testing of its efficiency would be somewhat troublesome for most platers there seems to be no reason why the complete agent should not be prepared and supplied as a ready-to-use plating chemical.

Discussion

Mr. S. C. Britton, after presenting the paper, referred to the desirability of agitation. He regarded the movement of electrolyte past the cathode as probably essential to obtain consistent results over shaped articles. Furthermore, if the plates were not given a light cathodic cleaning after plating they might be just a little duller than they should be and might develop a yellowish stain after some time.

The bath which at the time of writing the paper had been going for only a month or six weeks was now five months old, and was still giving fairly good results. He knew the difficulties which could arise; for example, the preparation of the addition agent required care. For the future an attempt would be made to simplify the preparation of the addition agent, and he was still examining the wood tars with the object of isolating the active material, or materials, from it. These things would make the process more commercially applicable.

Professor J. W. Cuthbertson (University of Nottingham) said that it appeared that for the first time a practicable process for the bright plating of tin seemed to be a possibility.

Tin had attracted the attention of the electroplating research worker for a number of years, and it was significant that only at such a recent date had such progress been made in depositing the metal bright. This was an indication of the difficulties involved.

The composition of the bath was interesting because it contained no cresol of phenol sulphonic acid. The authors mentioned that possibly their addition agent exercised some reducing effect, and he wondered whether or not this might perhaps in reverse shed further light on the straight matt plating bath and might ultimately show that in fact the cresol and the phenol sulphic acid were not really necessary. In this respect, if the addition agent acted as reducer, was it likely that its degradation products in due course might have a deleterious effect in the bath? Insufficient work had as yet been done to show whether or not that would be so, but no doubt the authors would have it very much in mind.

Up to the present the only practical method of obtaining tin bright was by flow brightening. That process worked extremely well particularly in the production of electro-tinplate, and he did not think that the new bright plating process would readily create a revolution in the electro-tinplate field. However, as regards thicker deposits, and particularly fabricated work, the coatings were often too thick to be flow brightened. The new process seemed to be a method of producing a coating which was not only as good as but perhaps superior to a flow brightened coating, and superior from the point of view of finger-marking. It was gratifying to know that so far as the tests have gone there was nothing to indicate that the corrosion-resistance of the bright tin was inferior to that of matt tin.

The point was made in the paper that the bright coating was considerably harder than matt tin, but no explanation was offered. He assumed that it might be due to a combination of a fine-grain structure and possibly something occluded in the deposit; had the authors considered or investigated the effect of low temperature on this coating? All were aware of the allotropic change which could occur in tin; fortunately it did not happen frequently, but from time to time trouble was caused in tin coatings which had to operate at low temperatures for long periods of time. This change could be suppressed by impurities; had the authors yet investigated whether or not this bright tin coating was less susceptible than the ordinary matt coating? If it were less susceptible, that might be an interesting and important point, suggesting its use in certain applications where there was a danger, however slight, of this change occurring.

Effect of Temperature

From the paper he noticed that the brightening effect disappeared when the temperature of the electrolyte was raised to 50° C. He wondered whether or not the fact that the coating was a hard coating meant that it was metallographically in some respects different from a matt coating. For instance, did this particular bright tin anneal as readily as a matt pure tin. If not, was this effect of temperature possibly associated not solely with a change in the electrochemical conditions of deposition but possibly with the fact that at 50° C it was possible for the coating to anneal, whereas at ambient temperature it did not do so?

Brittleness in the deposits had been referred to, but there was no mention in the paper of possible internal stress. Normally stress in a tin coating would not be expected but if the deposits did not anneal so readily, was there a possibility of some stress in the coating, and had that been considered?

Finally, with regard to structure, he was not aware that the work to which the authors referred, carried out in France, had yet been published, but here again there seemed to be further confirmation of the theory that the bright coatings were bright because they had (*a*) a fine crystal grain-size, and (*b*) a preferred orientation. He would be interested to read the French paper, because he would like to know more about the particular orientation found in the deposits. Tin was one of the few examples of a metal deposited every day which was non-isotropic. It had a tetragonal structure, and he would like to know how that fitted into the picture with regard to the orientation which was observed.

Dr. F. A. LOWENHEIM (Technical Adviser, Metal and Thermit Corporation, U.S.A.) asked whether the authors had had their samples long enough to show that they did not develop brown stains on ageing, as he had seen that happen with previous samples of bright tin. Also, he said, what was the solderability going to be after six months?

He thought that bright plating would not take the place of flow brightening, possibly because the porosity of the electroplated tin was no better than that of ordinary matt tin. In flow brightening the main object was to reduce the porosity.

What would be the minimum deposit which would be bright? This was of considerable interest, because he thought that most platers would still prefer if possible to plate their tin from stannate. Could they plate in the stannate bath and then finish up with this bright bath?

Mr. W. MARCHAND (Standard Telephones and Cables Ltd., London) asked whether the authors had carried out any quantitative work on the comparative throwing power of the ordinary stannous-sulphate bath and the bright bath? In connexion with the cathodic cleaning required afterwards, was there any other means of carrying this out, for example, by immersion treatment, and, in particular, how would this affect barrel plating work?

Had the authors found that the adhesion of the bright coating was in any way inferior to that of the matt coating, and had they had any blistering troubles in exposure tests?

With regard to the staining which developed, had the authors done any work with their chromate-phosphoric post-treatment to see whether or not this assisted in retaining the brightness of the deposit?

Mr. C. WATTS (Wilmot-Breedon Laboratories, Solihull) asked whether the brightness which was lost on heating returned on cooling, or did the heating permanently affect it? The work of the Tin Research Institute on tin-nickel seemed to indicate that the authors' work might not be exclusively stannous, and that encouraged him to ask if the tarry matter had been shown to have brightening or levelling powers in other electrolytes such as zinc or nickel.

Mr. S. W. BAIER (British Non-Ferrous Metals Research Association, London) said that he had

Personality Spot

ROY HAMMOND, B.Sc., A.R.C.S., F.R.I.C.

President, Institute of Metal Finishing

ROY ALBERT FREDERICK HAMMOND was educated at Aske's (Haberdashers' Company) School, New Cross, London. On leaving school in 1918, he entered Government service as a Probationary Assistant Analyst in the laboratories of the War Department Chemist, Royal Arsenal, Woolwich. When the war-time temporary staff was dispersed in 1919 he obtained a post as Assistant Chemist to Venesta Ltd., Silvertown, manufacturers of plywood, metal foils and collapsible tubes.

He resigned this appointment in 1924 on being awarded a London County Council Senior Science Scholarship to the Imperial College of Science and Technology (Royal College of Science) and graduated with Honours in Chemistry two years later. He was elected Associate of the Royal Institute of Chemistry in 1926 and to the Fellowship in 1950.

On leaving the Royal College of Science he again entered Government service in what was then the Research Department, Woolwich (now known as the Armament Research and Development Establishment). After a short period in the Analytical Laboratory, Mr. Hammond was transferred to the Electrodeposition Section of which Mr. D. J. Macnaughtan was then in charge. The late Mr. A. W. Hothersall became head of the section on Mr. Macnaughtan's appointment in 1933 as first Director of the newly-formed Tin Research Institute and Mr. Hammond himself succeeded Mr. Hothersall as head of the Section in 1950, the position which he still holds.

Mr. Hammond joined the Electrodepositors' Technical Society (now the I.M.F.) in 1927 and has been active in its affairs for over twenty years. He has been a member of Council since 1946 and a Vice-President since 1949 and was first Chairman of the London Branch Committee. He was nominated by Council to represent the Institute on the Productivity Team on Metal Finishing, which visited the U.S.A. in 1950 to study American methods and he served as Secretary of the Team.

His external interests are wide and include music, carpentry, metal work, drawing and photography.

seen bright tin deposits in the early 1930's, but from that time onwards there seemed to have been no real development until this paper. It had always seemed to him that the addition agents used in acid tin baths were essentially different from those used in nearly all the other baths. Most were of the type of dispersion rather than an organic agent which dissolved in the bath. *B*-naphthol was not soluble in an aqueous solution, and it was kept there with gelatin. In the early work crude cresol was used and was allowed to sulphonate in the bath for smoothing out in the electro-refining. The early workers could not maintain stability, and they did not know what it was all about.

Authors' Reply

Mr. S. C. BRITTON, replying, referred to Professor Cuthbertson's comment that the tar condition was probably exercising some preservative action against oxidation in the bath. This was very likely so, and in the ageing of the bath deleterious products might be formed in due course. That was one of the reasons why the authors were anxious to get as rich as possible tar fractions, to avoid putting into the bath materials about the action of which nothing was known, but which might prove in the long run to be disadvantageous.

With regard to future applications of the bright plating process, it was certainly true that the process should have a considerable advantage over flow brightening matt deposits, in that it would not be restricted to the thickness of the deposit. Mr. Lowenheim also referred to applications and wondered what the prospects really were for tinplate production. It would be altogether premature to suggest that the process was sufficiently reliable to be made a continuous process as yet, but it was certainly possible to obtain, in the rapid flow conditions which prevailed in producing strip-tinplate, very good and reliable bright deposits. It would not be a good thing, however, to introduce a coating of rather inferior ductility.

Professor Cuthbertson wondered why the coating was hard, but answered the question himself, because that was almost certainly due largely to the extraordinary fineness of the grain, and possibly also to the occluded material. Whether the occluded material would affect the performance at low temperatures was not known, but it seemed unlikely. Materials which were active in repressing the development of the allotropic transformation were metallic impurities such as bismuth and antimony, and, so far as was known, a non-metallic material was most unlikely to be active.

Professor Cuthbertson and Mr. Watts referred to the effect of heating the solution. It would be unwise to go too far in discussing this point. No evidence of advantage was found in heating, and the authors did not particularly wish to produce a heated bath if it was not necessary. It might be possible, however, by adjustment of the electrolyte, to get bright deposits at elevated temperatures; this was due, it was thought, to plating conditions and not to the annealing of the coating.

The authors had no indication of stress in the deposits, and the brittleness which had appeared from time to time required the application of stress manually to demonstrate it. The orientation of the tin crystals, as found in Professor Trillat's examination of the tin, was going down from the 112 plane, and Professor Trillat suggested that for deposits on copper epiaxis between the twins and traces of the cuprous oxide lattice still left upon the copper must have made some contribution. He pointed out that the fit between the tin and the cuprous oxide lattice was extremely good. He was now making further experiments with deposits on other metals.

Mr. Lowenheim asked about darkening on ageing. If no post-cleaning is given to the deposits some darkening would occur fairly rapidly, but deposits have been kept for nearly a year, and they still seemed to retain their original appearance. Solderability with ageing had not been tested, but clearly should be. Since the deposits seemed to be not inferior to other tin coatings in developing an anti-corrosive surface during storage, he thought that the solderability would be satisfactory. Deposits as thin as 0·0001in. were fairly bright, and such deposits had been plated over stannate tin and over tin-zinc and a satisfactory bright finish obtained.

Throwing Power

Mr. Marchand asked about the throwing power of the two solutions. The authors had made no rigid comparison of the bright tin and the ordinary sulphate bath, but from plating experience it should be much the same. It was true that post-cleaning cathodically was an additional troublesome process. A few experiments had been made with alkaline cleaning in an attempt to combine alkaline cleaning with passivation by using an alkaline-chromate solution, but the results so far had not been particularly hopeful. The adhesion should not be inferior to that of the matt coating.

Mr. Watts referred to the effect of heating. The solutions did give a bright coating when cooled down again, so that it would be possible to choose high temperature conditions and still get bright coatings. The authors had not tried the effect of the tar addition agent on other passive electrolytes, but the Americans had made a few experiments, and obtained slightly promising results with one or two.

He was glad to hear Mr. Baier's remarks. The idea of getting bright tin deposits had always attracted a good deal of attention, and Mr. Britton said that he believed that the reason for the failure over the years to get satisfactory results with this

sort of process was that people had not chosen either the tarry material or the dispersing agent sufficiently well. He thought that the authors' contribution lay in having laid the foundations at least of the means of making a choice of materials which gave hope of successful results.

Abstract No. 9:

The Adhesion of Surface Coatings

By W. D. May, N. P. D. Smith* and C. I. Snow**

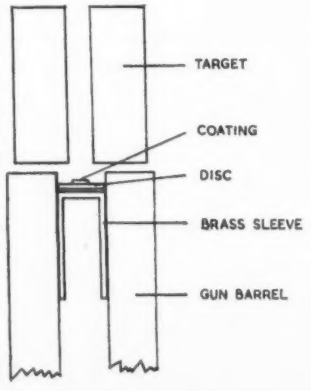
PROGRESS in the study of the adhesion of organic surface coatings has been hampered by the lack of a satisfactory method of measurement. Most of the traditional methods of paint industry measure quantities that are not primarily dependent on adhesion, but rather on a combination of mechanical properties of which adhesion is only one.

The ways in which it is feasible to measure adhesion can be classified as follows:

1. Methods in which a force is applied to the free surface of the coating, such as by pulling a test-piece cemented to the coating after it has dried.
2. Methods in which the coating is peeled from the substrate by forcing a wedge-shaped tool between the coating and substrate or by exerting a pull on a strip of fabric that is incorporated in or attached to the coating.
3. Methods in which a force is applied to underside of the coating through the substrate, such as by applying air pressure through a hole in the substrate.
4. Methods in which the substrate is subjected to a large acceleration such that the coating is detached by its own inertia.

Methods 1 and 4 can be expected to provide a measure of the force of adhesion, but those in 2 and 3 can, in general, give only a measure of the work of adhesion since the distribution of the applied force is not generally known. The paper describes and illustrates test methods of each of the four general types mentioned above and discusses their relative advantages and disadvantages.

* *I.C.I. Ltd. (Paints Division).*



Diagrammatic section of I.C.I. method of measurement of paint adhesion by rapid deceleration of bullet, whereby small area of paint is subjected to high inertial forces.

Discussion

DR. G. E. GARDAM (Aluminium Laboratories, Banbury) said that to find out something about the adhesion of a paint film, the most obvious test was to pull it off in a direction normal to the surface; the difficulty was to get hold of it. The availability of Araldite cold-setting adhesives, had made possible the method which the authors had first mentioned and this had been used with some success. Dr. Gardam had developed a simple apparatus, similar to that of the authors as he was more interested in coatings on thin sheet metal.

That test had indicated ranges of adhesion very similar to those obtained by the authors, but the gun test which they had described seemed particularly elegant.

However, the practical definition of adhesion, as viewed by the customer, was not necessarily resistance to a direct pull-off but to bending and twisting and pulling about generally. This difficulty arose particularly where lacquer systems were applied to thin aluminium sheet which was afterwards used for sauce-bottle caps and so on, where the lacquer coating with the metal received considerable deformation and in some cases came off, and then the question was, was it a failure of adhesion in the sense of direct pull-off?

Deformation of a piece of lacquer-coated metal gave rise to a force tangentially along the interface, deforming the lacquer, and four different results were possible, depending on the bulk properties of the lacquer and not so much on the adhesion. If the lacquer is brittle but has good shear strength it cracks, but remains there. If it is brittle and has poor shear strength it tends to come off as a complete film. If it is ductile the whole system moves together and no damage is seen. In the fourth case, if it is ductile but has poor shear strength, the join may be seriously weakened, but that may not be apparent until long afterwards.

In these four cases the direct pull-off adhesion was not necessarily in question.

In the first three cases mentioned, if the paint adhesion is measured on undeformed metal by a direct pull-off test, it has about the same value, but the use of a tensile test gives failure in two cases and none in the third.

Dr. Gardam asked whether the authors thought that the results which they were going to get with these tests were going to be particularly useful in the ordinary judging of customer complaints.

MR. N. D. P. SMITH (*in reply*) said that he

accepted the points which Dr. Gardam had made. The test was not intended to be passed directly on to the customers as the customer was not interested in how adhesion worked, but the paint manufacturers had to design paints which would stick, and therefore had to know the mechanism by which they did stick.

It was obvious that the force which broke the adhesion bonds was in general transmitted through the film itself. It was therefore necessary to study the whole mechanics of the paint film; not merely the bond, but also the effect of the other mechanical properties. The value of the test, was that it assisted the investigation of adhesion proper, that is, the specific adhesion force which holds the paint to the substrate, which must be the prime consideration, modified, of course, by the mechanical properties of the coating.

MR. G. H. JENNER (Schori Division, F. W. Berk and Co. Ltd., London) said that the paper had mentioned solder, and it was interesting that the values of adhesion obtained with solder on steel were much higher than those with paint. Two factors seemed to be involved; one was that on all-metal coatings there was a certain amount of elasticity which perhaps was not the case with paint films, and the other concerned the speed of application of the first load. Therein might lie an explanation for the difference in adhesion of the solder between the two tests, which could be bound up with the deformation of the bullet. The deceleration of the bullet, especially at the higher velocities, was so rapid that the elastic deformation must have a 'profound effect on the surface. As the bullet struck, it would bend to some extent, and, while there was a very large force pulling the coating off one way, there would be a minute force in the other.

Apparently, the use of sandpaper on the test specimens makes a difference. From his work on peen plating, he was naturally interested in very clean surfaces in order to promote cold welding, since that is the way in which the coating was held on. It had been found that if the surface was shot-blasted and then peen plated, the coating would adhere but then if the sample was bent the coating flaked off. If, after shot-blasting, the article was given a short immersion in dilute acid, there was no visible change, but the adhesion was profoundly increased. Removing the surface film and then doing something quickly on top, it made a very big difference to the adhesion.

Two things appeared to be very important. First, the massiveness of the bullet, to keep the deformation to a minimum. Second, with a critical adhesion test, as that one was, the provision of a really clean surface. As the method became more and more refined, so the surface had to be more and more refined, particularly for metal coatings.

MR. N. D. P. SMITH (*in reply*) agreed that there was quite a number of uncertainties connected with the deformation of the bullet at the very high impact velocity, and methods were being sought of decreasing the deformation and so on. The problem was not an easy one. There would be some advantage in using thicker bullets than the relatively thin metal sheet in use at the time as a substrate. Thin sheet made it possible to carry out very many more tests than if a very much thicker bullet were used, which would be difficult to stamp out of sheet and would require the use of specially made little pellets, which would then have to be individually coated.

The thickness of the bullet seemed to have relatively little effect on the values obtained, provided it was not too thin.

Dr. S. WERNICK (Consultant, London) asked how the test stood in relation to the more commonly-used methods of measuring adhesion, such, for example, as the cross-hatch test.

MR. N. D. P. SMITH (*in reply*) said he would not presume to criticise any of the older tests as they all bore some relation to the performance of the paint in practice, because that was the way in which a paint film failed. It was scratched by something being rubbed against it, or it was kicked or abraded, and so on. From a practical point of view, that was probably the easiest way to test the article; but care should be used in calling such tests adhesion tests. They were not adhesion tests; they were, although it might appear to be a subtle distinction, tests to see whether or not the paint would stick, which was a different matter. Whether or not a paint would stick depended in practice not only on adhesion but on the rest of the mechanical properties of the film.

The purpose in designing that apparatus was not in the first place to show whether or not a paint stuck, but to show what effect the true adhesion, the bond between the metal and the coating, had on the ability of the paint to stick. It would help manufacturers to design paints which had a better ability to stick, but it would not be the ultimate test of whether a paint would stick or not.

Inter-coat Adhesion

MR. R. J. BROWN (The Austin Motor Co. Ltd.) said that the paper made no mention of the possibility of using the test to determine inter-coat adhesion. No doubt it would be difficult to apply the test for that purpose, but it was a problem which concerned most of those who had responsibility for the application of multi-layer coatings.

MR. N. D. P. SMITH (*in reply*) said that the method could be used for inter-coat adhesion in certain cases.

In the deceleration test, the force varied from 0 at the free surface to a maximum at the bottom.

If the force available at any interface was greater than the adhesion, failure would take place. In a specific case with a good primer but weak intercoat adhesion failure would depend on the adhesion being roughly two-thirds or smaller of the adhesion on the substrate. The chance of the coat next to the substrate failing in adhesion was greater than one up.

Abstract No. 10:

Advances in Spray Booth and Oven Design

*By A. L. Newcomb**

THE need for producing better paint finishes more economically has demanded higher operating efficiency from spray booths and ovens. In addition maintenance costs and operating costs are being considered more closely. Increased production has resulted in increased overspray, and water-wash booths must now be designed to wash efficiently larger amounts of overspray from the air. Exhaust volumes must be determined more accurately and the larger users of paint spray booths have become more insistent that the water-wash chambers of a booth exhaust the amount of air for which they were designed. The efficiency with which the water-wash chambers scrub the paint overspray from the paint-laden air is also receiving more attention and in the U.S.A. some of the larger users have set up allowable limits of paint pigments in grains of solids per 1,000 cu. ft. of air which may be discharged.

The redesign of spray-booth water-wash chambers by some of the manufacturers in the U.S.A. has been extensive in order to cope with the increased paint loadings and to meet new test and performance requirements.

In addition to washing the paint pigments out of the exhaust air the wash chamber should be designed to minimize the possibility of any deposit of paint pigments inside the washer, and accessibility to all interior surfaces and the ready removal of all water piping, together with adequate inspection doors are features of newer designs.

There is a growing tendency to conduct performance tests during production, and "drifting" of overspray is becoming an acute problem due to the wider ranges of colours being sprayed on a single conveyor line. Air distributing baffles, consisting of two layers of perforated metal, have proved a positive method of regulating the flow of air to a booth from overhead.

An important design feature that is most helpful in attaining satisfactory "balance" of supply air and exhaust air is the installation in a spray-booth of the pressurized vestibule. Conditioned air is supplied to an enclosed vestibule at the entrance to the booth and air is also supplied to a vestibule at the exit end of the booth, *i.e.*, between the booth and the oven. Air is supplied to the vestibules to create an air velocity of 100 to 150 ft. per min. through silhouetted openings, thus preventing the entry of dust at the entrance end and the entry of oven heat at the exit end.

In all sludge disposal systems, water treatment to control the paint waste plays an important role, and the problem of sludge disposal in the more recent installations is now more acute than the former maintenance problem of removing paint accumulation from the spray booth water-wash chambers. The ultimate in sludge-disposal systems has not yet been attained, and there seems to be no relation between the initial cost of a system and its overall efficiency.

One advance in the maintenance of high-production spray booths is the introduction of conveyorized floor grating and conveyorized floor plates, particularly in down-draught spray-booth tunnels.

Two modifications of the dry-type of spray booth have recently appeared on the American market. One of these incorporates the use of inexpensive throw-away type paper filters to trap the paint overspray and the other which appears to be adaptable to continuous production, employs inexpensive flame-proofed felt-type cloth media for the same purpose.

In the U.S.A. direct gas-fired heating systems are being employed extensively to heat the "make-up" air required in spray-booth installations.

The major change in oven design in the U.S.A. has been the use of larger air volumes in the heat re-circulating systems to accomplish faster and more even drying. The main source of heat is gas, including natural gas. The heaters are of the direct gas-fired scroll type. Most oil-fired heaters are, however, of the indirect type, and have limited air capacities. However, in Holland, a large indirect oil-fired heater with an air capacity of 25,000 cu. ft. per min. is operating very successfully.

In ovens which employ large air volumes, the air recirculating ducts and the exhaust ducts are generally installed at the top of the oven so keeping the walls and floor free from dust-accumulating surfaces. The ovens are treated with a film to trap any dust that might be present in the circulation system and the bottoms of the ducts are often hinged to assist cleaning.

Long ovens using large air volumes are usually "zoned", and in many instances ovens are being more closely checked to see that they perform up to design standards.

The use of catalytic combustion systems for conserving heat is in an embryonic stage, but a number of these are being installed, both in the U.S.A. and France. Another interesting develop-

* *President, Newcomb-Detroit Company, Detroit, Michigan.*

ment is that of exhaust fume incineration, where the exhaust air is heated to about 1,400° F for ½ second and is then discharged into the moisture dry-off ovens.

Discussion

Mr. I. WALKER (Head of Laboratory, Briggs Motor Bodies Ltd.) said that it was stated in the paper that in down-draught spray booths, practice had established that exhaust air volumes for down-draught spray tunnels ranged from 1,500 to 2,000 cu. ft. of air per linear foot of tunnel.

It was also stated later that excessive exhaust volumes actually increased paint overspray by drawing the paint mist away from the work. Could the author be more specific on that point? Was it possible to say at what exhaust air volume the condition was reached of increasing overspray, and what volume would secure freedom from overspray, in a booth with a conveyor operated at 10 ft. per min. with a 3-ft. space between bodies, which was the example cited in the paper.

Reference had been made to the necessity of ensuring that the correct air volume was supplied to the spray booth at the proper temperature, and that the make-up air was evenly distributed to the booth. Could the author give some information on the tolerances that were possible in the latest booths used in the U.S.A., and how evenly could the temperature and humidity be controlled?

In connexion with the pressurized vestibule and the need to ensure that sufficient air was supplied to the vestibule to restrict dust from entering the booth and, at the exit end, to restrict oven heat from moving into the booth it was often surprising to find that the entrance doors to those vestibules were badly fitted. A great deal of money was spent to obtain dust-free air which was passed to a chamber which itself was not dust-proof. The answer might be that there was sufficient air pressure in the vestibule to ensure that the dust would not enter; but, because it was not possible to tell the air exactly which way to go, there were parts of the door surround which invariably let in air and dust.

Air filters were another problem. Could the author give further details of the types of air filter which were suitable? He had said that in some factories different filters were used in different parts of the same plant. Had sufficient work been done on those comparisons to enable some specific recommendations to be made?

The dry type of spray booth was referred to in the paper. Had he any information on the advisability of considering this type of booth in future for the spraying of motor bodies?

With regard to ovens, for moisture drying re-circulating direct-fired gas ovens were recommended, and these were, of course, in use in this country. One difficulty appeared to be that in recirculation a considerable amount of water vapour was also recirculated, and when the oven was closed down that high humidity tended to corrode bodies which were left in the oven. What was the author's experience of that in American production? It appeared to be American practice to use direct-fired gas recirculating ovens for final enamel stoving. Did the author consider that that method of stoving was satisfactory for the high sulphur-bearing gas which would have to be used in the U.K. for heating?

The author had mentioned the installation of a large volume air heater with a stainless-steel combustion chamber on an oven in an automotive plant in Holland, and gave the very impressive tolerance in temperature of ± 4°F maintained throughout the heat zone. Did this installation of large volume air heaters show a marked trend for the future, or were the operating costs too high to justify the expense? For the more normal type of recirculating ovens what was the control of the tolerance, and how did the control obtainable on direct, indirect and steam ovens compare? Some further information on catalytic combustion systems would be of interest.

Finally, it would be interesting to hear the author's thoughts for the future. He had claimed that the manufacturers of paint finishing equipment were keeping up with the rapidly expanding use of paint finishes for commercial and domestic products. Did he consider that the plant manufacturers would follow the developments in paints, or would they be able to lead those developments with revolutionary changes in oven design and so on, as for instance, the combination of infra-red and convection heating of ovens. Claims had been made on the Continent that the quality of the finish can be improved in that way, but there seemed to be some doubt about that.

Specification for Booths

Mr. A. H. COWAN (Drummond Patents Ltd., London) envisaged the possibility that some body might be set up to consider the drawing up of some kind of specification to cover the passing out of booths, and that the specification should be built up on three main subjects.

First, the primary object of a booth was to give good operating conditions to the workers and draw away the overspray so that they did not breathe it. Second was the provision of clean air, so that when it was vented to atmosphere it would create the minimum nuisance, the ideal being no nuisance at all. The author had mentioned a figure of 3-5 grains per 1,000 cu. ft. That was quite possible,

and had been attained with water-wash booths over many years in practice. It appeared to be the view in the United States that air giving figures of that kind could be recirculated back into the shop, which would mean a very great saving of heat; but it was unlikely that the U.K. Factory Inspectorate would ever agree to that.

The third point was maintenance, with which was closely bound up the question of disposal of the sludge, and the simpler that was, the better. It was to be hoped that as a result of this meeting it might be possible to initiate a study into the means of working out a pass-out specification in that field.

Flash-off Time

Mr. R. M. C. LOGAN asked whether, with the speeding-up of stoving times, American practice had been able to reduce flash-off time, or was that holding them up on total stoving time?

Mr. G. J. H. CARROLL (Carrier Engineering Co. Ltd.) said, on the testing of water-wash chambers, that it was an excellent achievement in the U.S.A. to arrive, as the result of tests, at a figure of 3-5 grains of solids per thousand cubic feet of exhaust air as the desirable maximum for the contamination of the exhaust, but he wondered whether even that was not too dirty.

He agreed with the author that there was still a great deal to be learnt about sludge recovery systems. It would appear that advances in the efficiency of sludge recovery would reduce maintenance costs more than any other advance in spraying. To dig out a tank involved overtime and week-end work. If the paint sludge could be conveyed outside the paint shop and removed automatically during working hours it would be a major advance.

A wide variety of filters were in use, from electrostatic down to simple fibre glass in two layers. There did not seem to be any very noticeable difference in the cleanliness of the finished job in either case. What was much more important was the cleanliness of the body before it entered the booth.

Dry-type Booths

The CHAIRMAN asked the author for more information on the dry type of booth. In the U.K. it was becoming more and more difficult to obtain water in the first place and to get rid of it when contaminated in the second. Quite a large volume of contaminated water was produced by the water-wash booth.

He went on to suggest that there was no synthetic enamel paint shop which was free from trouble with dirt, and questioned whether the industry was providing clean air of a much higher standard than necessary and taking far too much dirt into the booth by its methods of application and of cleaning, and by the general way in which paint shops were organized?

Author's Reply

Mr. A. L. NEWCOMB said that the question of the velocity of the air to carry away overspray was a difficult one. A single operator spraying with a gun at a certain pressure and with a certain type of paint would require a lower velocity to carry his overspray away than two or three men operating in a confined area.

With regard to the temperature in spray booths and how fine a control of temperature was possible, he recalled that two summers ago he visited Dagenham when there was an outdoor temperature of about 66° F and it was very humid, and men started walking out of certain operations on the plant. The spray booths there operated at 60-65° F. Spray booths in America operate at 75-80° F, so that U.K. spray men presumably would not work in American spray booths, and vice versa, so human nature came into the matter.

How closely the temperature of an air supply system could be controlled was a very important matter indeed. The manufacturers of equipment went to a great deal of trouble in the engineering of an air supply system which had to operate, much of the time, under varying climatic conditions and yet permit a rise of only between 3° and 6° F in the booth. To engineers, a system to give control within about 3° was quite a task when using 5,000 cu. ft. of air per min. There were not many manufacturers who would tackle the problem of doing that.

Humidity presented no great problem; with the equipment available it was not difficult to control the humidity in a system.

Air filters were a very considerable subject. The dust problem was one of the biggest with which the industry had to deal, and all engineers were looking for a combination of filters which would do the job properly. There was need for a wide variety of filters for different applications.

With regard to the use of direct-fired gas on final coats, naturally the contents of the gas had a great deal to do with whether it could be used or not. The use of a gas such as coke-oven gas was not desirable. U.S. practice was to use manufactured gas, with filtering systems to filter out the impurities. For moisture drying ovens and so on, however, a wide variety of gases of varying cleanliness could be used. For final coats clean gas was essential.

He had been asked whether he could control indirect oil-fired heaters to within $\pm 4°$ F. The particular case mentioned in the paper dealt with a large quantity of air, and air in large quantity did not give high peaks, and therefore he would say

that with an indirect oil-fired heater of this type he could control to within $\pm 4°$ in ovens.

He agreed that it was difficult to take out the vapours and odours prevalent in enamels and enamel overspray, which would cause trouble in any attempt to recirculate spray booth air, even after it had been cleaned.

It had not been possible to shorten the flash-off time from the time the product was sprayed until it reached the oven as with the newer paints now in common use that flash-off time was necessary.

Comments had been made on the question of whether 3-5 grains per thousand cubic feet was a good yardstick for the cleanliness of the air. The idea of testing spray booths in this manner was not much more than two years old, and he agreed that as time went on it should be possible to make the mechanism so efficient that an even tighter standard could be maintained.

The equipment described could readily bring the metal up to heat in 4½-5 min., but with the larger air volumes it was possible to have a lower head. To try to bring the temperature up in that way with lower air volumes would definitely require higher temperatures, which might conceivably cause difficulties.

The dry booth had great possibilities. The basic factor was the cost of the filter cloth, but as the demand increased the cost would come down, although it had not yet reached an economic level. In some cases, however, the dry booth could already justify itself by eliminating water piping costs, sewage piping costs and so on.

The very strong point made by the Chairman about cleanliness in the paint shop touched one of the most important problems facing the industry. Speaking on behalf of the manufacturers, Mr. Newcomb said that they all tried to build equipment with a good air filtering medium in it and to design equipment to keep the dirt out of spray booths, but generally the housekeeping in painting departments was very lax. Much could be done by good cleaning and housekeeping programmes. Large amounts of money were spent on very good equipment, and then the workers were allowed to bring in dust on their shoes and their clothes and defeat the operation of the equipment.

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Abstract No. 12:

Airless Spray

*By T. Cowlard**

THE airless method of paint spraying combines the use of heat with hydraulic pressure obtained from a suitably designed piston-type pump giving pressures of 300 to 600 lb. per sq. in. The method reduces many of the disadvantages of conventional methods, such as overspray, necessity for water-wash booths, etc. The temperatures used in the airless method range up to 160° F for cellulose lacquer and 180° to 210° F for other materials, enamels, synthetics, epoxy resins, etc. When the paint particles are ejected at high velocity they meet air resistance which causes individual particles to subdivide.

The paint is heated to a point where the lower boiling solvents have appreciable vapour pressure (3 to 25 lb. per sq. in. above atmospheric pressure) and the sudden release creates rapid expansion and a further division of the atomized paint particles. In this system the solvent in the paint acts as an atomizing agent.

The equipment required consists of a ram pump, which sends the paint under pressure *via* a 4-kW heater to the spray gun. Unused paint is returned by a circulating pump to the heater. Fresh unheated paint is passed, together with the heated paint, through the heater. The spray gun is a special type with a fluid metering control, and inter-changeable nozzles. These nozzles are manufactured with accurate spray angles, the correct angle being obtained by means of a precision machined slot. "Telflon"-lined hose is used because of the high operating temperature and pressure. Adequate safety devices are incorporated in the system, which, by careful selection of the spray nozzle for the work being processed, can produce a film of predetermined thickness.

The advantages of the process include: reduction in paint loss (up to 40 per cent.); reduction in health hazards; strong exhaust systems not required; improved finishes. As regards the latter, a heavier film can often be achieved in one operation, and paint adhesion is often higher than with other methods.

In the majority of cases standard paints can be sprayed provided that 80 per cent. fast solvents and 20 per cent. slow solvents are used. Some hot-spray formulations are not satisfactory with the airless method because the solvent evaporation rate is too slow.

The scope of the system is wide. For example, it can be used for spraying large pieces of machinery on site, and because of the lack of rebound and fumes it is possible to spray in enclosed spaces. Fan widths of up to 2 ft. 6 in. are possible, effecting great saving of time, particularly on ships' hulls, etc. In the U.S.A. the motor-car industry is steadily changing over to the airless system, both for hand and automatic spraying. The system is also being used in the U.S.A. for applying aircraft finishes.

The system gives a more speedy flash-off when

* *T.C. Spray Finishing Systems.*

spraying lacquers, and chlorinated rubbers are being applied successfully with the elimination of cobwebbing.

Abstract No. 13:

Automatic Spraying

*By J. Webb**

AUTOMATIC paint-spraying machines fall into five classes, *viz.*: (1) the traversing type of automatic; (2) single-turntable type; (3) batch-turntable type; (4) spindle conveyor with following spray equipment; (5) other specialized types.

The first type consists of guns traversing across a moving belt-type conveyor carrying the work, with the guns spraying downwards on to the work. This is the most reliable method of obtaining an even finish on a flat or near-flat surface. It should be realized that the number of coats applied depends on the fan width of the guns, and a traverser is preferably mounted above the roller-type conveyor so that the guns can operate between two of the rollers. In this way the conveyor does not get painted when the work does not completely cover it. The traverser and conveyor are usually combined with an oven or some drying arrangement which in turn can be coupled to other processes.

With the second type of machine, which can be electronically timed, the components are mounted on a revolving chuck and coated by means of a fixed spray gun. This is the most inexpensive form of automatic spraying, and is becoming very popular, particularly when fitted with an electronic time switch operated by a foot pedal. The time switch is responsible for opening and closing a solenoid-operated air valve controlling the spray guns. Articles of many shapes can be handled on this type of machine, and it is often possible to spray the inside of, for example, a tin, by using one stationary gun.

The batch-turntable semi-automatic machine is similar to the single-turntable type except that the turntables are fitted with a hundred or so spindles. The whole turntable is made detachable for subsequent drying of the sprayed articles.

The spindle conveyor with following spray equipment is completely automatic, the work-pieces being loaded at one end, painted, stoved, cooled and off-loaded at the other end. The conveyor consists of spindles at 4-in. centres and travelling at about 8 ft. per min., the spindles being paired together and carried on two-wheeled skates. A sliding carriage carries the spray gun forward for about 3 in. before it springs out of engagement to move back to the next spindle. The articles to be sprayed are carried on the spindles by means of chucks designed to suit the article being processed. After spraying the work-pieces are stoved in an infra-red oven.

* *Aerostyle, Ltd.*

Discussion

Mr. T. COWLARD (T.C. Spray Finishing Systems, London), in presenting the paper on "Airless Spray", added: "In the past twelve months we have gone a considerable way in determining the correct size of orifice for a given performance of material. At the moment we are standardizing on six sizes of orifice. By this method of metering—the metering orifice is very accurately machined—we are able within reasonable limits to guarantee not only the flow rate but also the film thickness, but we are not always able to guarantee the exact fan width."

Mr. H. J. MASON (Vauxhall Motors Ltd., Luton) said that there was no other commodity sold which could be made or marred by application more than paint. Those who have had the task of evaluating paint over the years had realized how easy it was to obtain completely contradictory results purely on account of the method of preparation or application of the panels. The authors had described types of equipment claimed to be capable of producing a standard thickness of paint film, properly applied; if the claim was substantiated it would be a great step forward.

Economically the airless spray should have something in its favour, as, he said, compressed air was very expensive to generate. However he was worried with regard to the pumps and metering devices described, because he felt that there would be difficulty in metering the correct amount of paint to give the desired paint film thickness. Furthermore, how was the equipment to be kept clean and could the author give some idea of the ease of changing colour and of the difficulties which would have to be met to ensure cleanliness? If so, what was the scope of the paint film thicknesses which could be applied by the metering device, taking into account the fact that different paints required different film thickness.

Reference was made in the paper to the use of warm cellulose at 600 lb. per sq. in., and that required careful attention to fire regulations. He hoped that Mr. Cowland could convince his local factory inspector or fire chief that warm cellulose solution at the above pressure was a safe medium to use in a paint shop!

The equipment described in Mr. Webb's paper appeared to be fairly simple, a straightforward back-and-forth type of spraying, or spraying at a specific and fixed angle. Mr. Mason thought, however, that the author ought to have said more about the automatic spraying of difficult contours, because the equipment needed for that type of application was very complex. It was necessary to

simulate the movements of the human arm and wrist to obtain the correct thickness over the metal contours, and that was not easy. Had Mr. Webb had any experience in that direction, and could he give some tips with regard to the mechanical construction of the bearings and the lubrication of the various joints on the machine which had to perform these rather flexible movements? He also asked Mr. Webb for information on the speed of colour changes.

Concluding, Mr. Mason asked what were the approximate relative costs of the pump and the jets and the compressors needed for airless spray on the one hand, and the cost of applying the same amount of paint with normal compressed air and a normal compressed-air gun on the other?

Life of Spray Nozzles

Mr. J. N. T. Adcock (I.C.I. Ltd., Paints Division, Slough) said that he believed that one of the uses of airless spray would be in the application of undercoats and fillers and the pigments used in these products would have quite an abrasive action, particularly at pressures of 500 to 600 lb. per sq. in. In the early days of airless spray brass nozzles were used but were not entirely satisfactory, and he thought that they had now been supplanted by sapphire jets or jets of hardened tungsten carbide. Could the author give some indication of the life of the jets in actual service?

Problems of Atomization

Mr. C. I. Snow (I.C.I. Ltd., Paints Division, Slough) commented that the demands for improved and thicker films and films of higher gloss called for a careful balance of solvents in the paint when it reached the object to be painted. With present methods of spraying there was a need to allow for the rapid loss of solvent as the paint broke up into drops. When he first heard of the airless spray, he thought that it removed one difficulty in improving paint systems, but he found that it was necessary to use the solvent to break up the paint into drops.

Problems of breaking liquids into drops were met with in other industries, and particularly in combustion engineering, and a good deal of work had been done there in designing special nozzles to break up quite viscous liquids into fine drops. Very briefly, the work which had been done showed that it was necessary to get the liquid into a thin film in some way or other before surface tension would break it up. The new spray gun which produced a narrow jet of liquid was said to break the liquid up into drops by stretching it out, which at once presented problems when it was desired to use new resins, which might form fibres.

There was one form of nozzle, however, which had been used quite widely. It had a conical shape, and there were baffles incorporated which caused the liquid to swirl round at very high speed inside the nozzle just before it came out. This meant that when it was ejected its centrifugal force made it fly out into a cone. The liquid in the cone was very thin, so that the minimum amount of work had to be done in breaking it into drops. It seemed to him that airless spraying, with its high pressure, was an obvious application for this type of swirl spray nozzle, and this might make it possible to use less solvent.

Mr. R. M. C. Logan said that he had had about nine months experience of airless spray, with the snap action gun, and a brass nozzle wore out in about a day, or in three days at the most. Other types lasted considerably longer, but were much more expensive. If the metering orifice wore as quickly as the nozzles, it raised the question of how accurately the paint could be applied. Mr. Cowlard had said that improved adhesion was obtained but he strongly recommended him to quote users as saying that, and not to fall into the trap of himself saying it.

Mr. Webb referred to the build-up of paint on nozzles of automatic guns. His cure was an interesting one, *i.e.*, a method of cleaning the nozzle; but had anyone bothered to design a nozzle with an improved aerodynamic flow, so that paint did not build up on it in the first place?

Mr. G. Greenfield (Steel Equipment Co. Ltd., West Bromwich) said that Mr. Cowlard had made what seemed to him to be an extravagant claim in saying that he could guarantee the film thickness. Surely the question of operator application came in here? Did he consider that it was easier to operate the gun in airless spraying than in the conventional forms of spraying?

The author also referred to the wide use of this method in America, but there was a great diversity of opinion, inasmuch as steam spraying was extremely popular in America. Could he give the reasons why he considered airless spraying to be more satisfactory than the other method?

Recently he had received some literature about a conventional type of gun which held the atomized paint in a curtain of air, and it was stated that this could save 40 per cent. of the overspray. Could Mr. Cowlard give information about this?

Mr. A. H. Thomas asked Mr. Cowlard how consistent were the results which were obtained with nitro-cellulose and vinyl lacquers. It had been reported recently that very inconsistent results were obtained with these lacquers.

Mr. L. Gaiger (The Aerograph Co. Ltd., London) asked: (i) was there a normal trigger variation to control the paint flow with the airless method? (ii) did the spray pattern remain constant during a normal period of operation? (iii) What was the nozzle wear factor? (iv) Although

the visible overspray was reduced, what was the position with the solvent?

MR. L. WOOD said as a user of airless spray for about twelve months, he might be able to answer one or two of the questions which had been raised on Mr. Cowlard's paper. The user was naturally interested in economics, not so much in the cost of the plant, but in how soon the plant would pay for itself. His company's plant, at a most conservative estimate, had paid for itself twice in the first year.

The question of changing colours had been raised. As users, the first problem required to be solved was how to minimize colour change, even though the colour could be changed in 15 minutes.

On the question of sapphire and other nozzles, his company had done a wide range of tests, starting with brass. Brass lasted for about half an hour of actual spraying time, sapphire suffered 0·0006 in. wear in 5-hours' spraying time, and tungsten-carbide 0·00025 in. in 73-hours' spraying time. Taking cost into account, the tungsten-carbide nozzle was by far the cheapest.

With regard to spray pattern, with the original gun provided by Mr. Cowlard, he had had considerable trouble, but with the new metering gun he was able to meter quite successfully, and it practically eliminated any trouble with the primers.

On the question of economics, he had no figures comparing ordinary spray with airless spray, but he had compared brush painting with airless spray. On one job alone, comprising about 2,500 sq. ft., 70 man-hours were saved compared with brush painting. There was also a great difference in the variation in thickness of the coating. With brush painting there was a 0·0045-in. variation in thickness. That might sound like bad workmanship, but it was probably standard practice in many firms. The variation with airless spray was 0·0005 in.

MR. I. WALKER (Briggs Motor Bodies, Dagenham) said that he was alarmed at the thought of having to use the Teflon-lined, stainless-steel-braided hose to which Mr. Cowlard had referred. The weight of the hose must be considerable, and this meant that the operator would have more difficulty in maintaining consistency, unless it could be linked with the automatic method. The cost of the hose was also likely to be high. By this method of spraying, the paint arrived at the metal surface hot, and that gave a higher gloss, but he suggested that there were other and more consistent ways of obtaining a higher gloss.

A new gun which he had had demonstrated to him (and which he thought was the one referred to by a previous speaker) gave another way of cutting down this paint rebound, which seemed to be the main advantage of airless spraying. It was a question of surrounding the sprayed pattern by a cone of air, and a 40 per cent. saving in paint was claimed. So far he had had very limited experience of this gun, but it did help on some difficult jobs in spraying inside motor vehicles. It was so simple that he could not understand why it had not been thought of before.

On automatic spraying, though this was a very difficult field he was trying to reproduce the motions of the human hand, wrist and arm. He was thankful at the moment that he had still some skilled sprayers left, as the idea of fixing an automatic traversing device to do plain sheets and then having a man to watch it working, did not represent much of an advance.

Authors' Replies

Mr. T. COWLARD, replying, said that he would like to thank Mr. Wood for answering some of the questions from the point of view of the user. Mr. Mason was concerned about wear through the abrasive action of paints. It was admitted that a certain amount of wear would take place in any moving part when in contact with paint, but what his company had done was to minimize this wear and reduce it to economic limits. By the simplified design of the pump, replacement of the parts which wore could be effected very quickly. With the most abrasive types of red-oxide paint the seal should last at least three months and as regards the mechanical seal, this took five minutes to change.

With regard to the wear problem, the velocity of the paint and the erosion rate could be high, but it had been proved that tungsten carbide was the best material available. Wear, however, was obviously proportional to the type of material being used. Taking red oxide as an example of a material which was bad in this respect, when painting large fabricated structures the nozzle should last at least 500 to 600 hours. A longer life than this would not be obtained with a conventional compressed air-gun, with a paint flow at a much lower velocity.

With regard to the metering device, tungsten-tipped needles and a tungsten insert in the fluid orifice were being fitted apart from the actual metering nozzle.

Mr. Snow had referred to paint formulation and solvent control, but experience had been that with certain materials this solvent problem was not so acute as originally both paint formulators and users believed. The "tails" which Mr. Wood mentioned were caused in some cases, though not in all, by the mechanics of the spray gun. With the vinyls and epoxy resins in particular it was common to get these tails. With the high-viscosity materials and high surface tension materials the solvent vapour pressure was critical, but with 50 per cent. of the standard materials it was not necessary specially to formulate the solvent.

Mr. Logan had made some comments on the control of film thickness, but Mr. Cowland had not meant to convey the impression that a hand-operated airless gun cut out hazards due to the operator. If the gun was being used at a variable rate, variable film thickness would result, whatever was done about the mechanics for metering a given amount of material in a given time. The airless method used in conjunction with automatic spraying would bring nearer the ultimate in the matter of uniform film thickness. In his experience the average film thickness with the airless method had been about 0·0015 in. per pass of the gun. That was based on a vinyl-base material at a viscosity, at 70° F ambient, of 45-50 seconds. That viscosity, under heat, was reduced sometimes to as low as 16 seconds. With some materials, heavily pigmented and a low boiling solvent it was possible, by repeated passes of the gun, to put on a film up to 0·005-0·006 in.

Hot Cellulose

Mr. Mason had mentioned cellulose at 600 lb. per sq. in., but he believed it was correct to say that a cellulose mixture in an enclosed hydraulic circuit, even if all the solvents were taken to a temperature above their boiling point, would not form, without the introduction of oxygen, an explosive mixture. Already he had had long discussions with the local inspector of factories on these points. If a compressed air source of supply on the cold material was being used he would expect trouble, but it was possible to suck out of an open vessel containing paint with no pressure behind it.

Mr. Walker had commented on the weight of the hose, but, Mr. Cowland said, it was half the weight of the standard fluid hose used in ordinary practice.

With regard to this cone gun, he was astounded that somebody should come forward with the suggestion that this gun was new. In fact it was used in 1933, and was then produced in Leipzig, in Germany. It was turned down in those days purely on mechanical grounds, the reason being that the earlier gun consumed 75 cu. ft. of air, and was thus expensive to run. Whether a gun had now been produced to give a sufficient pressure of air at a very low consumption he did not know.

Mr. Gaiger asked about the control; there was a fluid metering control on the airless gun, and this had been found necessary for the reasons stated. It was possible to control the velocity of the material through the nozzle; the pattern was very constant. Because of the higher velocity of the material through the nozzle there was no build-up around the nozzle which might have caused distortion or mal-dispersion of the paint; however, it was constant only so long as the wear was not outside the limits laid down.

In no circumstances would he advocate a man working in a solvent vapour. The dispersion of solvent vapour depended on the area in which the spraying operation was taking place and the provision made for its dispersion. In the paper he asked paint manufacturers to avoid the use of toxic solvents where spraying took place with no form of air stream.

The CHAIRMAN: Mr. Cowlard, replying to Mr. Mason, said that hot cellulose at the high pressure in question is safe because there is no oxygen present to form an explosive mixture. He is quite right, but nobody worries about it when it is still in the system; the point is what happens if a burst occurs, and there is plenty of oxygen to cause trouble. That may be a consideration when we hear Mr. Cowlard the next time!

Mr. J. WEBB, replying, said that with regard to contour spraying, his experience was mainly of the articles illustrated and largely concerned getting over the troubles by careful positioning of the gun, or guns if several were used, and making use of the spray pattern. It was quite possible to coat satisfactorily a large variety of shapes with the fixed spray gun, without having to move it, and then there was no bother about lubricating the moving joints, which might become coated with overspray. Careful positioning of the gun could reduce overspray enormously and reduce paint wastage when compared with that caused by a hand-spray operator, who might wave his gun about in an unsatisfactory way. With a fixed spray gun, the gun could be focused on the job and the overspray reduced by a very substantial amount.

Spraying Interiors

The inside spraying of articles could very often be done most satisfactorily without the necessity to move the gun inside and up and down and in and out of the article. The gun need not necessarily have a round spray; a fan-shaped spray could be used. A point about spraying the inside of articles with the fixed gun was that there was a certain amount of spray-back which coated the nozzle. His company's guns did not suffer from it badly, but when spraying the inside of an article the spray-back would coat the nozzle base to a certain extent. To make sure that the nozzle was kept operationally clean, on the completely automatic plant he had arranged for a little gun to spray the nozzle face to keep it clean. Automatic spraying machines, because they handled such a colossal production, could be dirty if badly adjusted. If it was desired to spray only part of an article, a stencil was very often necessary, and that could give a great deal of trouble unless it was automatically cleaned, so that on this machine a self-cleaning stencil was incorporated. It was a disc which revolved sandwiched between two revolving brushes. When the brushes revolved in opposite directions the disc

turned and the article being sprayed was passed behind the disc, so that the disc cut off the spray. The disc was then immersed in a trough of solvent to keep it clean.

Colour changes were a simple matter on the type of machine shown. Two sets of spray guns, two sets of containers for the paint, and two foot pedals, were required; it was as simple as that.

The tips did not wear out quickly, and he would regard 500-600 hours as quite a short life. Tungsten-carbide tips were available and these lasted much longer.

Abstract No. 11:

The Application of Finishing Materials at Controlled Temperatures

*By John Muirhead**

THE use of heat to assist in the deposition of surface coatings permits the use of finishing materials with much higher solids content than would be possible with other methods, resulting in a substantial reduction in the amount of solvent required. By achieving temperature control the viscosity of the finishing materials can be so regulated as to obtain optimum efficiency in surface coating. The lack of viscosity control is often the reason for a high percentage of such defects as "orange peel", overspray, and low film build.

Many improvements can be obtained by hot spraying. The only true valuation of paint cost is the area which can be covered at a given dry film thickness per gallon. With the hot spray process various degrees of film thickness can be obtained and overspray reduced. When applying material by the hot-spray process the solvent loss is greater since the material comes out of the gun near the boiling point of the solvent. This condition is desirable provided that sufficient solvent remains in the deposited coat to effect correct flow out. The application of a full wet coat is the object to be achieved in almost all finishing processes and most spray operators have a tendency to deposit a wet film which closely approaches the sagging point. In using hot-spray methods the "wet point" is reached sooner because of the correct solvent balance and higher solids content, thus there is a greater margin of safety. There is one ideal temperature at which any given paint performs best and at that temperature the paint flows out smoothly with the least "orange peel". The hot spray process gives a less porous and more even film structure, because less solvent is present. In addition the reduced solvent content gives less shrinkage in the deposited paint film and the filling properties of the coating are proportionately improved. "Blushing" is also prevented by the hot-spray process.

A further advantage is that an operator can apply, in one stroke of the gun, the equivalent of two coats, and as a result drying time is considerably reduced, particularly with cellulose lacquers where the one coat often replaces two or three coats applied by cold methods.

The hot spraying of synthetics offers considerably less difficulty than that of the lacquers, some of which will spray drier and will not flow out as well as others. With low-viscosity lacquers, vaporization may take place inside the heating units when the pumping system used for circulation introduces new lacquer, and as a result "spluttering" may produce an uneven coat. A pressure feed tank eliminates this trouble which does not occur with heating systems which do not employ circulation.

The characteristics of some lacquers may restrict their use where flow rates of over two pints per min. are required, and if such lacquers are "pushed" to such rates then a tendency to "bubble" may be caused. Although it is the chemists' job to produce a lacquer with the minimum tendency to "bubble", such defects can be caused by high shop temperatures, draughts, etc. In order to apply lacquer at elevated temperatures successfully the operator must be familiar with the film thickness of the cold lacquer system he wants to replace and a lacquer with the proper characteristics chosen. He must also know how far the flow rate can be increased without causing "bubbling".

Abstract No. 14:

Aerosol Spraying

*By E. V. Sharpe**

AEROSOL spraying utilizes fluid expansion to promote atomization and then a gas stream to help carry the particles so produced to their destination. While the volatile constituents of all paints have a vapour pressure, most of the solvents used develop insufficient power, at room temperature, to either atomize the paint or to carry the particles. With a mixture of suitably formulated paints or lacquers, and a freely miscible fluid (the propellant) having the necessary vapour pressure, it is possible under certain conditions to cause both atomization and propulsion.

In an aerosol paint container, therefore, a mixture exists of the appropriate paint with a predetermined volume of propellant fluid which breaks up the paint into small globules and vapourizes on release to the atmosphere. The propellants used in the vast majority of aerosols are the fluorinated hydrocarbons, known in the U.K. as "Arctons". While the cost of these materials is relatively high, they

* *The Aerograph Company Limited.*

* *Robt. Ingham Clark and Co., London.*

have few of the shortcomings associated with other low-boiling liquids such as methylene chloride and butane. The mixing proportion of paint to propellant is normally in the region of 1 : 1 but can vary between 2 : 3 and 3 : 2. In general the aim should be to keep the viscosity of the mixture low while keeping the solids content as high as possible.

Two types of surface coatings are most popular for aerosol packages; these are solution types such as nitrocellulose or acrylics, and alkyds of medium to long oil-length.

The behaviour of fluorinated hydrocarbon propellants in the presence of air-drying alkyd media can be likened to that of aliphatic hydrocarbons under the same circumstances with the consequent danger of precipitation or gelation if carried too far. Solvency must be catered for in the original formulation of the medium or vehicle by using aromatics to a greater extent than normal. The selection of solvents must also take into account evaporation rates, as once atomization has taken place, many of the usual problems of spray application apply. When a satisfactory wet film has been applied by the aerosol method the normal characteristics of flow and solvent release are required.

Whereas the fluorinated hydrocarbons are tolerated in limits by alkyds in general, they are definitely diluents to nitrocellulose and many other solution-type media. To obtain the required solvent strength, therefore, the volatile content of the initial lacquer formulation must normally consist entirely of true and latent solvents, but no diluents. A large number of successful nitrocellulose aerosol lacquers use methyl isobutyl ketone as the main solvent. A second choice is butyl acetate. To assist in absorbing moisture, either butyl "Cellosolve" or "Cellosolve" are added.

To prevent settling of the pigment in the container, antisettling agents are used. The use of a steel ball or a pebble inside the container also assists in breaking up and redispersing settled pigment.

Care has to be taken in filling containers to prevent boiling, and an efficient yet cheap valve on the container is required. A precision-made removable valve is probably the best solution but this type of valve is not yet available in the U.K. The majority of containers are either of tin-plate or aluminium, the former being the cheaper to produce.

Before use, a paint aerosol should be well shaken, and the valve checked for cleanliness. The most satisfactory method of spraying is to apply thin coats so as to build up the required film thickness.

Although aerosols are not intended for spraying large areas, their use makes it possible to spray materials which normally could only be poorly applied or even not applied at all without full professional equipment.

Discussion

Mr. J. N. T. Adcock (I.C.I. Ltd., Paints Division, Slough) said that the application of paint by spray was one of the most wasteful operations it was possible to imagine, and any step which would overcome this wastefulness was obviously a good one to take.

All were convinced of the merits of hot spray method as it was a sensible thing to do, rather than add thinner, to raise the temperature and so drop the viscosity of the paint. He would like more information from Mr. Muirhead, however, on the mechanics of getting a steady output of paint at the optimum temperature so as to produce controlled film thickness. Could he give information on the merits of the circulating and non-circulating types of hot spray plants? In the paper the author referred to a hot spray unit which coated with 200 gal. of paint per hour. That seemed rather a lot. He presumed that a number of guns were used, and he would be grateful for some more information about this, and particularly about the economics of the large hot spray units.

Overspray

It was stated that overspray was less with hot spray than with cold. Mr. Adcock said that he did not think that all those who had had experience of the hot spray method would support this, but perhaps the author could say precisely how much was in fact saved. In the paper on spray booth design there was reference to testing the efficiency of spray booths; could Mr. Muirhead say what he had done to test how much less overspray there was with hot spray than with cold?

Mr. Sharpe had given a very complete and admirable summary of the present position with Aerosol. On the subject of propellants he mentioned that the Arctons had advantages over any others, and although this was probably correct, there ought to be something to be said for butane, propane and butane-acetone mixtures, chiefly from the point of view of solvency and the compatibility characteristics. Referring to the nitrocellulose lacquers, Mr. Adcock said that he thought that the lowest viscosity was obtained with the highest total solids, which was what was required, by using acetone as the solvent, and, compared with the compositions which the author quoted, there might be advantages in reducing the amount of medium "boilers" such as the butyl acetates and replacing them with acetone.

The biggest problem of all with aerosol finishes was that of pigmentation. It was necessary, as the author had said, to have very fine grinding, and there had to be a very careful choice of pigments, which would restrict the colour range considerably.

So far as settling was concerned, help could be given by anti-settling agents; but in a market where aerosols might be used to the greatest extent, *viz.*, the retail one, there were likely to be long storage periods, and he felt that some of these anti-settling agents, after the aerosol had been in store for six, nine or twelve months, might make the ultimate dispersion of the settled pigment even more difficult.

On the subject of valves, he would like to learn more about the removable spray heads which the author mentioned, because, Mr. Adcock said, his company had had a good deal of trouble with valves sticking or being scored with paint. He thought that perhaps paint was not really a suitable material for an aerosol container, and it reflected great credit on the makers of valves and on the formulators of paint that they had produced such a satisfactory job. The tendency, however, for the nozzles to gum up and for the valves to stick constituted a very real drawback.

Mr. Sharpe in his paper referred to a 1 : 1 ratio of paint to propellant. Mr. Adcock would like to see some plasticiser present, or the use of a high-viscosity nitro-cotton, otherwise there might be a little cracking.

Heat Transfer

Mr. T. COWLARD (T.C. Spray Finishing Systems, London) said that the objective of hot spraying was a constant temperature at the source of supply, namely the spray gun nozzle; how was the heat transfer efficiency determined? Paint had a specific heat, and this had to be known for the calculation of the heat transfer efficiency. Mr. Muirhead had mentioned plants of 200 gal. per hr. Had he calculated the amount of energy necessary to create the *x* B.Th.U. required to raise the temperature of the paint in that volume? How costly would such a piece of equipment be?

Mr. A. E. MANTELL said that Mr. Muirhead had made sweeping claims for the savings which could be made by hot spraying. Was it his opinion that hot spraying was advantageous only where heavy coats are wanted, or was it possible to effect any saving in thin coats? Had the author any thoughts on using heat as a viscosity constant? He was thinking more in terms of circulating systems or even pressure pots, where it might not be desirable to use true hot spray. Had he also any experience of raising to a known temperature, and thereby achieving a known viscosity, a paint which might be suitable for either cold spray or hot spray?

Could Mr. Muirhead give figures to indicate what the savings due to reduction in overspray were?

Mr. I. WALKER (Briggs Motor Bodies, Dagenham) commented that so far as spraying operations were concerned, he thought that the future probably lay with a combination of automatic electrostatic and hot spraying techniques. How much of each had still to be determined, but he felt that hot spraying would probably play a useful part in further development. For assembly and cutting out wet sanding in motor body production, which consumed a considerable amount of labour, the first need was for a better metal condition, which he thought could be achieved, and better scratch-filling properties from the paint materials, combined with better flow, giving a more even coat with scratch-filling properties.

Scratch Filling

Recent tests which had been conducted with this in mind showed that by using the hot spraying technique with materials of much lower viscosity than was usual it was possible to obtain better scratch-filling properties and to limit considerably the amount of wet sanding. There were still limitations; one concerned the equipment, and he hoped that some of the equipment manufacturers would take the opportunity to enlarge on what had already been said about the application of finishing materials at controlled temperatures and give some idea of the limitations of the different types of hot spraying equipment which were available.

The type had to be matched with the type of operation. For instance, spraying one type of material continually in large production did not demand heating of the paint in the actual pipeline; it was sufficient to use a comparatively simple heating operation and then pass the hot paint through the pipeline; as the paint was moving continuously, it did not have a chance to get cold. In other cases, however, this might be a serious limitation, because the paint in the pipeline remote from the heater would have a chance to cool down, which meant each time spraying was stopped, emptying the pipeline before it was started again, or starting under unsatisfactory conditions.

He would welcome further information about the types of equipment available from the various manufacturers, and more information about the difference between circulating and non-circulating types of hot spray unit.

One advantage, he felt, of hot spraying was that for colour coats it was usually necessary in spraying enamel to do two separate spraying operations over the body, which meant two lots of overspray and twice wasting paint in that way. If it was necessary to make only one application it would save paint material, apart from its other advantages.

Mr. C. I. SNOW (I.C.I. Ltd., Paints Division, Slough) said that in introducing his paper, Mr. Sharpe had suggested that it would be interesting to make a comparison between the two methods of paint application, and there was one point to which

both authors referred, *viz.*, that both methods of application were restricted in the thickness of single coat which could be applied in one coat by troubles with bubbling. The hot spray method described by Mr. Muirhead used air to push the paint out and to disperse it. The paint was hot while being dispersed. Air was not particularly soluble in paint systems, and was even less soluble when it was hot, so that in this case it would be thought that the formation of bubbles must be primarily a matter of air entrapment. Mr. Sharpe's aerosols used Freons, which, though perhaps not very good solvents, were at least compatible with paint, so that the bubbling in his case might well be due, as he suggests, to the Freon not having time to get out.

Mr. Muirhead said that the maximum flow rate which could be used was two pints per minute, while Mr. Sharpe advised keeping the aerosol container at least 10 in. away from the work. Perhaps, therefore, the authors would give figures for the maximum thickness in microns of wet film which could be applied by their respective methods without running into bubbling troubles.

Authors' Replies

Mr. JOHN MUIRHEAD said that Mr. Adcock had referred to the wastefulness of spray painting in general. Although this might appear on the surface to be true, was it not true to say that much of the waste was the fault of the operator? He thought that a great deal had still to be done in the training of operators. Much of the overspraying and waste could be controlled by the correct use of equipment.

Mr. Muirhead emphasised the fact that a hot spray unit was not a "box of tricks". All that was being done was to produce at the gun a viscosity at which the paint manufacturer desired the material to be applied. The hot spray unit itself was merely a means of producing sufficient heat to achieve the viscosity required. He could therefore reply to questions concerning the amount of heat required for any given film thickness or viscosity by saying that the amount of heat required was not a stated amount, but merely the amount which would produce the required viscosity at the gun.

With regard to circulating and non-circulating types of equipment, perhaps two points in favour of the non-circulating type were that from the material container, material was being drawn at its original viscosity, and when the operator started spraying the material was left in that state; it was not circulating through the unit or being used at all until the operator opened his gun. With circulating units, the pump might have a capacity six to ten times that of the spray gun. It was not possible to say that material would circulate once round the system and then come out of the spray gun; it might circulate all day before it left the spray gun.

By referring to 200 gal. per hr. in the paper he was describing multi-gun units of large heating capacity in the form of multi-kilowatt heaters for large production purposes.

In stating that the amount of overspray was less he could give any figures, but with the reduction of the pressure in the hot spray method it should be apparent that the overspray would be reduced. If the pressure was reduced from 75 to 50 lb. per sq. in., there would definitely be a reduction in the overspray.

Constant Temperature at Supply Source

Mr. Cowlard had asked how the constant temperature at the source of supply was obtained. All that was required to be done was to achieve a certain viscosity at the gun. Whether the material was heated by hot air, steam, hot water or even tea did not matter so long as that viscosity at the gun was obtained, and thus tests were necessary to establish the correct conditions.

Hot spray equipment could be used with conventional materials, *i.e.* materials intended for cold spray application, and the subjection to heat of those materials provided the benefit of being able to use lower pressure. He could give no details in answer to the question about experience in raising materials to a known temperature; in the hose there was a drop in temperature, and it was necessary to maintain the temperature right up to the gun, and this was done by circulating along the hose the heating medium.

It is reasonable to suppose that paint spraying systems were under discussion at this small conference gathering which includes (left to right) *Mr. J. Muirhead* (*The Aerograph Co. Ltd.*), *Mr. G. J. H. Carroll* (*Carrier Engineering Co. Ltd.*) (standing), *Mr. A. L. Newcomb* (*president, Newcomb-Detroit Co.*), *Mr. T. B. Hardcastle* (*Carrier Engineering Co. Ltd.*), *Mr. R. B. Mitchell* (*managing director, The Aerograph Co. Ltd.*) *and Mr. Jakobinski* (*Newcomb-Detroit Co.*)

Mr. Walker had referred to the use of hot spray at low viscosities. Mr. Muirhead had already said that that was done, and it was perfectly possible to use these lower viscosity materials with the hot spray unit. In his company's system of hot spray only the paint required was heated.

As regards bubbling, he was prepared to abide by what the paint techologists said about this. He thought that bubbling had a good deal to do with the solvent content of the material, and by using materials which had been formulated with a proper solvent content much of the bubbling could be overcome; however, he would like guidance from the paint manufacturers on that.

Mr. Snow referred to a flow rate of two pints a minute. No comparison could be made on this basis. With one of the units which the author had in mind a flow rate of about 32 fluid ounces a minute was common, but that was, generally speaking, above the average capacity of the normal spray operator.

Flammability of Propellants

Mr. E. E. V. SHARPE, replying to Mr. Adcock, said that butane and propane or butane-propane mixtures were being used, and he believed that on the Continent they were used quite extensively. He objected to them from the flammability point of view. It was possible with some materials to keep the flash-point above the figure, in the cellulose regulations, of 73° F, and so to keep the propellant outside that limit and have something which was safe for storage and did not come under the cellulose regulations. There were already a good many regulations about the use of aerosols, and should anything go wrong there would no doubt be many more. That was the main reason for considering the use of non-flammable propellants, in spite of the fact that they were considerably more expensive. As an example, an aerosol might contain 6 fluid oz. of lacquer and 5 to 6 oz. of propellant. With Arcton the propellant might cost 1s. 2d. or 1s. 3d. per pack, but with butane 3d. or 3½d. There was therefore a strong incentive to use butane, but the question of flammability would override it in many cases.

Mr. Adcock's next point was the use of acetone and the other ketones as solvents for the lacquers. It was necessary to use acetone with certain of the solution-type materials whether one wanted to or not, but then the question of flashpoint flammability came up. The tendency had been to keep the flashpoint as high as possible, consistent with the material being suitable for doing the job which it was supposed to do; but he agreed that acetone and other ketones could be and were used.

As regards the removable valve which could be cleaned, there were two examples of that in the exhibition, both of American origin. He did not know of one made in this country yet, but he believed that there were plans for making the valve head removable so that at least the orifice could be cleaned.

On the question of plasticisers, the formulation for a nitrocellulose clear lacquer given in the paper was not intended to be complete and a plasticiser would have to be added.

Mr. Snow asked about the limitation of the wet film thickness per coat. The limitation arose from the disposal of the propellant in the time elapsing between the aerosol orifice and the surface to be coated. If the ambient temperature was low, evaporation was retarded, even of a propellant ejected at well above its normal boiling point, and the tendency was therefore to build up too wet a film on the surface being coated. If that film was wet because propellant was trapped in it, the propellant would find its way out and might do so very drastically. If the distance between the aerosol and the surface was too short an over-wet film would be produced. The best way to overcome this was to build up the dry film thickness required by applying a thin wet coat and then another thin wet coat and so on. The general use of aerosols was as a rectification tool or to touch up small work by the amateur, and the amateur had usually plenty of time to adopt the above method.

On the question of wet film thickness, it was possible to put on one of 50-60 microns, depending entirely on the material, but assuming a solid content of, say, 50 per cent., that should result in a dry film of about 0.001 in., which was reasonably easy to achieve with the correct technique of application.

Storage of Aerosols

He would like to mention some of the techniques in the use of an aerosol and in storage, etc.; one was to build up a film to the required thickness in very quick passes, spraying a light film on, allowing it to flash off and then putting on another coat, and so on, until the desired effect was produced. In much of the rectification work on a motor car, for example, it might be necessary to build up films several thousandths of an inch thick to make good a surface, and to try to do that in one coat was to ask for trouble.

Another necessary precaution concerned storage and the temperature of storage. On the label it normally states "Do not store in direct sunlight and store preferably at a temperature below 70° F". He knew of an aerosol exposed to the sunlight on the shelf of a car, and it blew up.

A Quarterly Survey of some of the Features in Finishing Literature from Abroad

by **SCRUTATOR**

ALTHOUGH they comprise an important part of our motor exports, commercial vehicles tend to be looked upon as the cinderella of the industry as far as developments in finishing are concerned. It is interesting to learn, therefore, that because of its light-weight and favourable modulus of elasticity, magnesium is now being employed by American truck builders(1). In fact, there are now some 20–30 American firms building van, platform and flat-cowl truck bodies containing large amounts of magnesium. Although, originally, painting the magnesium was something of a problem, it is claimed that this has been simplified by the development of new cleaning treatments and primer coats. Perhaps most significant of all, sheets and extrusions are now coming from the mill with a baked primer coat already applied. This is yet a further example of the rapidly increasing tendency in America for both pretreatment and some painting to be carried out at the sheet or strip mill.

Turning to the more glamorous side of the motor car industry, a considerable amount has been published on the use of anodized aluminium for automobile trim on Continental cars, but, so far, comparatively little has appeared regarding American practice. Some details have, however, recently been given by Nixon and Kahler(2) regarding General Motors' practice. This includes a detailed description of the Cadillac Motor Car Division's processing practice for aluminium grille parts. It may be new to many that for General Motors the present trend towards brightened and anodized aluminium trim was started by the Cadillac Motor Car Division with the introduction of gold-coloured licence plate frames on their 1954 models. Since then, applications have increased so that on 1957 Cadillacs and Chevrolets, grille parts, emblems, scripts and trim moulding are either natural or gold-coloured anodized aluminium.

Chromium Plating

The results of two investigations involving chromium plating have been published. Both are of an exploratory nature and considerable further work will be required before it can be established whether they show commercial possibilities. They do, however, indicate some interesting fields for further development in the sphere of chromium plating. Hoover(3) has described some experiments on chromium plating over a Cronak surface film. He found that chromium could be easily electrodeposited on to zinc plated steel surfaces which had been Cronak treated and then heated, generally at 320° F for about one hour, before plating. Salt spray and humidity tests showed that the chromium deposit gave various degrees of protection, depending on thickness, but in one test 2,660 hours humidity testing at 110° F were required before blistering occurred. Zell(4) has described his attempts to deposit chromium from a trivalent bath. The basic constituents of the solution were chromium sulphate, ammonium sulphate, urea and formamide and although the operating conditions were extremely critical, by very careful control it was possible to obtain smooth, nearly bright and very ductile deposits.

Analysis

The analysis of electroplating and chemical pretreatment solutions is a subject which generally rceives little attention. It is somewhat surprising therefore to find that during the last three months or so two new analytical methods for electroplating solutions have been developed—both based on the use of E.D.T.A. Gabrielson(5) determined the lead content of five different lead fluoborate baths using an E.D.T.A. titration method and also gravimetrically by precipitation as lead sulphate. Good agreement between both methods was obtained; the maximum difference being less than 2 per cent. Without performing any quantitative determinations, it was found that iron and copper interfered to a certain degree.

Estimation of the amount of iron in chromium plating solutions is not normally a routine determination. According to Dettner(6) however, this can be performed by a quick, convenient volumetric method, again using E.D.T.A. 10 per cent. disodium—Benzcatechine—3,5—disulphonate is the indicator.

Odds and Ends

A further development in the sphere of electroless plating is the announcement of a process for the deposition of an arsenic-zinc alloy(7). Generally speaking, the equipment and method is similar to that used for electroless nickel and one of the

(*Continued in page* 178)

Overseas Review

(*Continued from page* 177)

principal uses for this coating may be as a substitute for the zincate method of plating on aluminium. It is claimed that any type of aluminium treated by this method can be plated with almost any other method without further treatment. The coating of metals such as steel, brass, copper, aluminium and, under certain conditions, stainless steel has been performed.

Doss, McHenry and Eisler[8] have published results on the effects of various chromic or chromic/phosphoric acid rinses as final treatments for phosphate coatings. Their reaction with thin phosphate coatings such as are used for paint bonding was one of the main points to be investigated. The experimental technique was to process steel specimens in each of five phosphating baths containing P^{23} as a tracer and make activity measurements before and after immersion in one of three chromic or chromic/phosphoric rinses. The percentage loss of phosphate coating was calculated from counting rates, and corrosion tests were performed to determine any loss of corrosion protection. They concluded that caution should be exercised when using such rinses for lightweight phosphate coatings, *i.e.* those produced from chlorate/nitrate or nitrate/nitrite accelerated zinc phosphate solutions intended for paint bonding. If any rinse is to be recommended for these, it should not have a greater concentration than 4 oz. of chromic acid per 100 gallons and 4 oz. of phosphoric acid per 100 gallons. Perhaps it is unkind to point out that this is exactly what has been used on thin phosphate coatings in this country for many years now. At any rate, it is always nice to have unbiased scientific confirmation of well established commercial practice!

References

(1) *Iron Age*, 1957, **179**, (6), pp. 106-107.
(2) *Plating*, 1957, **44**, (2), 156-160.
(3) *Product Finishing*, 1957, **21**, (4), 46-49.
(4) *Metal Finishing*, 1957, **55**, (1), 57.
(5) *ibid.* (3), pp. 56-57.
(6) *ibid.* (2), p. 67.
(7) *ibid.* (1), p. 56.
(8) *Organic Finishing*, 1957, **18**, (2), pp. 4, 6, 8.

VINYL PLASTIC-COATED STEEL

New Product for Decorative Use Now in Experimental Production in the U.S.A.

A HOST of interesting possibilities for new decorative effects are offered by a new product which combines the structural strength of steel with the rich decorative appeal of vinyl plastic in an almost limitless variety of colours and textures. This new development has graduated from the research laboratory stage and is now in experimental production at the United States Steel Corporation's Irvin Works in Dravosburg, near Pittsburgh.

The new material is produced by coating cold-reduced sheets with liquid plastic to produce an appealing, tough-surfaced sheet of great beauty and durability. It promises new decorative effects and long service in such equipment as refrigerators, radio and television cabinets, wall panels, motor-car bodies and trimming, stove and heater parts, railway-coach sleeping-car interiors, and office and home furniture.

In the manufacturing process, the steel is first surface treated to improve its bonding qualities and a specially compounded adhesive is then applied to the top surface. After heat curing and air cooling, the steel enters a coating chamber where the thermo-responsive vinyl plastic is applied to the adhesive-coated surface. Again heat is applied to solidify the plastic. Prior to cooling, the vinyl coating is embossed to obtain the desired design or texture. The finished product is then stacked by a magnetic piler.

The coating of this new material possesses excellent abrasion resistance and has good electrical resistance properties. It is unaffected by humidity and many chemicals. The coating also produces sound-deadening effects similar to that obtained by "muffle coating" car bodies and will have value in acoustical design.

The new product, which can be supplied in any standard colour, can be utilized for manufacturing many parts which previously required protective or decorative finishing after fabrication, such as car panels, radio and television cabinets, institutional and office furniture, appliances, kitchens, wall panels, etc. Elimination of the need for such post-fabrication treatment, coupled with the unique properties of the new coated sheet, will result in lower production costs, together with superior wearing qualities and decorative effects.

At present, due to the developmental nature of the operation, no definite annual production capacities have been established. The pilot-line facilities at the Irvin Works will produce 18-to 28-gauge sheets 24 to 52 inches wide. The semi-commercial product is being offered for sale on a development basis. There is no doubt that this new development offers many fascinating possibilities for designers and fabricators of sheet-steel products.

THE PHYSICAL SOCIETY EXHIBITION

A Review of some Prototype and Production Equipment of Immediate and Potential Interest to the Metal Finishing Industries

ONE of the attractive features of the exhibition staged by the Physical Society as compared with other more commercially-inspired exhibitions is the fact that so many of the exhibits are in the research or development stages and thus afford fascinating glimpses of the shape of things to come. This year's exhibition was held at the New Horticultural Hall from March 25 to 28 and, as usual, the Hall was filled to capacity by visitors throughout the whole period.

A number of the exhibits were of immediate or potential interest to the finishing industries and brief details of some of these are given here.

Coating Thickness Measurement

Much of the equipment on show was devised for the accurate measurement of various quantities, including the thickness of applied coatings. On the stand of **Nash and Thompson Ltd.,** Oakcroft Road, Chessington, Surrey, for example, was the commercial version of the B.N.F. Coating Thickness Meter, the research prototype of which was described in an earlier issue of this Journal.[1]

This instrument (Fig. 1) is a robust and easily-used meter capable of measuring non-destructively the thickness of electro-deposited coatings irrespective of whether or not the coating or the base metal is magnetic. Up to the present work has been concentrated on the measurement of nickel platings on steel, brass, and zinc alloys, in the range 0·5–1·5 $\times 10^{-3}$ in. to an accuracy of $\pm$ 15 per cent. The presence of a thin, decorative chromium deposit has no influence on the results, and means have been devised for determining the thickness of a copper undercoat if required. Indications are that the method can be used for the measurement of a wide range of electro-deposits and base metals and possibly also for the sorting of uncoated metals.

The instrument consists of two parts, a probe unit and a sensitive d.c. amplifier incorporating an indicating meter. The probe is essentially a heated copper body on which is machined a hemispherical tip, subsequently electroplated. When the probe is applied to a plated surface thermo-electric voltages are developed due to the juxtaposition of dissimilar metals and the difference in temperature of the various interfaces. The voltage generated between the probe body and the cold base metal, after amplification, is indicated on a meter and bears a simple relationship to the plating thickness which can be determined by comparison with known standards. For most purposes a chromium-tipped probe is used but when a copper undercoat is to be determined a nickel tipped probe is required in addition. Provision is made on the meter unit for the connexion and control of two probes.

The probe is designed to be applied by hand or through a simple jig with a steady pressure,

Fig. 1.—B.N.F. coating thickness meter

Fig. 2.—Industrial pH a nplifier

approximately normal to the surface of the test-piece, the minimum workable radius of curvature on concave surfaces being 0·5 in.

Manual application is satisfactory, a pressure of 5 lb. being required. In routine inspection and for highest accuracy a jig and lever system is preferred. A "cold" connexion to the specimen is required to complete the circuit, this being made by the special clip provided or through the base plate of the jig.

Once the instrument has been set up, all that is necessary is to clean the surface of the specimen, make the "cold" connexion, apply the probe and note the reading on the meter scale, which can be calibrated directly in plating thickness.

To set up the instrument two standard specimens, one for each end of the range of plating thickness, are used, The probe is applied to each in turn and the zero and sensitivity controls of the amplifier adjusted until readings are obtained at predetermined points on the scale. An hourly check should be sufficient to maintain satisfactory accuracy. Two small dry batteries are used in the zero adjusting circuit, each having an expected running life of 3,600 hours.

Detachable scales can be provided for calibration by the customer and one is available ready calibrated for the determination of certain proprietary nickel deposits.

pH Measurement

For the measurement and recording of *p*H, of growing importance in the metal-finishing industries, several instruments were shown and demonstrated by **W. G. Pye and Co. Ltd.,** Granta Works, Newmarket Road, Cambridge.

The "Master" *p*H meter, for example, is a new instrument of high accuracy and stability. It is direct-reading and mains operated. The effective scale of 0 to 14 *p*H is 11 in. long and full automatic temperature compensation is provided whereby the instrument can be buffered at one temperature and readings taken at another.

The model "H" industrial *p*H amplifier (Fig. 2) is completely new in design. Housed in a splash-proof robust, flush-mounting case the amplifier is sealed against corrosive fumes. The *p*H reading is presented on an 8-in. circular scale and full automatic temperature compensation is provided.

The new general-purpose *p*H meter is designed to provide the simplest possible operational arrangement and has only one control. It is mains operated and direct reading on a 5-in. scale covering *p*H 0–14. Temperature compensation is carried out automatically by the platinum resistance thermometer in the electrode system. Particular care has been taken in design and layout to provide the utmost reliability and ease of maintenance.

Ultrasonic Cleaning

The recent developments in ultrasonic generators have aroused interest in the possible advantages to be derived from the application of ultrasonics to

Fig. 3.—Ultrasonic cleaning equipment

metal-finishing processes. Up to the present time the most promising field of application has been in the cleaning of small intricate components, and a number of pieces of equipment devised for this purpose were on show.

For example, **Dawe Instrument Ltd.**, 99, Uxbridge Road, Ealing, London, W.5, exhibited their Type 413 ultrasonic cleaning equipment, which is the production version of the equipment shown at last year's exhibition. It embodies several new features, a more rugged construction and an improved cabinet design.

For this equipment (Fig. 3), pulsated operation at 40-kc/s. frequency is used. Cylindrical tank transducers are available with barium-titanate elements built into the base, the two sizes available being $5\frac{1}{2}$ in. dia. by 6 in. deep and $6\frac{1}{4}$ in. dia. by $7\frac{3}{4}$ in. deep, giving capacities of $\frac{1}{2}$ and 1 gallon respectively. Also available are immersible transducers for the introduction of ultrasonic cleaning into existing baths. All assemblies are of stainless steel but very highly corrosive liquids may be used by placing within the outer container a lining of plastic or glass. The output may be switched to either of two sockets, enabling washing and swilling to be used alternately. Cleaning occurs as a result of the rapid formation and collapse of vapour bubbles within the liquid. This cavitation effect cleans many assemblies difficult or impossible to treat by other means, and in almost all cases ultrasonic cleaning affords a striking saving in plant or time over other methods.

Another interesting development in this field was featured by **Radio Heaters Ltd.**, Eastheath Avenue, Wokingham, Berks. This was the new U.S.10 1 kW. output ultrasonic generator (Fig. 4) which is designed to give a reasonably high power output at a frequency in the 40/100 kc/s range.

For cleaning purposes this is claimed to be very much more effective than frequencies in the region of 1 mc. For example, it is possible to immerse 1 mc. transducers directly in water and operate them at full power without any deterioration of the silver facing. If, however, a similar transducer operating at a frequency of 50 kc/s is immersed in water, cavitation is so severe that the silver plating is stripped from the surface of the transducer within 30 seconds.

It is therefore necessary to protect low-frequency transducers in a casing which will resist cavitation erosion.

The biggest obstacle to the design of high-power low-frequency equipment was the low temperature at which barium titanate de-polarized. A low-frequency barium-titanate transducer operating at high power is extremely difficult to cool due to its great thickness. If any part of this transducer runs above 80° C it de-polarizes and ceases to have piezo-electric properties. This results in the rest

Fig. 4.—Ultrasonic generator with output of 1 kW.

of the transducer having to vibrate an inert mass. In practice this often results in shattering of the transducer.

These difficulties mean that a barium-titanate low frequency transducer can only be operated at extremely low power densities as it is impossible to get the heat away.

Another device to solve this problem is to operate the transducer in pulses. This, however, merely means that the transducer is only used for one-third of the total time, with the result that the cleaning operation takes three times longer than it would do if the transducer could be operated continuously.

Zirconate transducers can be operated without very serious loss of efficiency up to 250° C. Furthermore they do not de-polarize until 400° C. This means that they can be operated at quite high powers without any undue difficulty in cooling them.

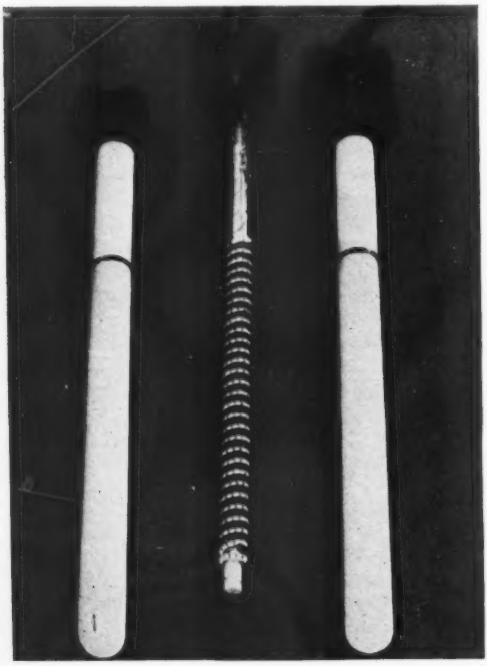
The Radyne US10 ultrasonic generator has been particularly designed to energize zirconate transducers but is, of course, equally satisfactory with barium-titanate transducers. In order to increase its flexibility, two frequency bands have been provided, the first at 50 kc/s and the second at 1 mc. The 50 kc/s band permits the efficient removal of large impurities while the 1 mc. transducer is particularly effective in removing light soils and fine grease films.

A section of the wide range of exhibits of **Mullard Ltd.**, Mullard House, Torrington Place, London, W.C.1, was devoted to ultrasonics. Of particular interest was a low-power ultrasonic cleaning apparatus operating at a frequency of 20 kc/s. The magnetostriction transducer is constructed of annular nickel laminations with a stainless steel or glass vessel of about 50-ml. capacity in the centre of the annulus. This cleaner has been developed for the high-quality cleaning of small precision components.

Vacuum Deposition

Examples were exhibited by **Edwards High Vacuum Ltd.**, Manor Royal, Crawley, Sussex, of applied thin film investigations carried out in their laboratory, while a new prototype vacuum coating plant, the Metrovac Type 18 was shown on the stand of **Metropolitan-Vickers Electrical Co. Ltd.**, Trafford Park, Manchester, 17. This equipment incorporates facilities for evaporation, sputtering and the "blooming" of optical lenses, etc. The working chamber is 18 in. dia. by 24 in. high, and controls, indicators, signal lamps, etc., are conveniently located on a sloping panel; the operating levers of the vacuum valves are brought out to the front of the cubicle which contains the pumping equipment and electrical gear.

Fig. 5.—Immersion heater and sheaths for alkaline solutions



The modern tendency to reduce as much as possible the size of electronic instruments has necessitated many developments in component design. For example, edge-cooled, high-temperature selenium rectifier units of remarkably small dimensions were shown by the **Westinghouse Brake and Signal Co. Ltd.**, 82, York Way, Kings Cross, London, N.1. These rectifiers are capable of operating at full load, when mounted on a chassis, at 100° C. Heat is removed by conduction from three of the edges of each square element, the fourth being left for connexions. The complete units are resin sealed. This company also showed some new process copper-oxide rectifiers.

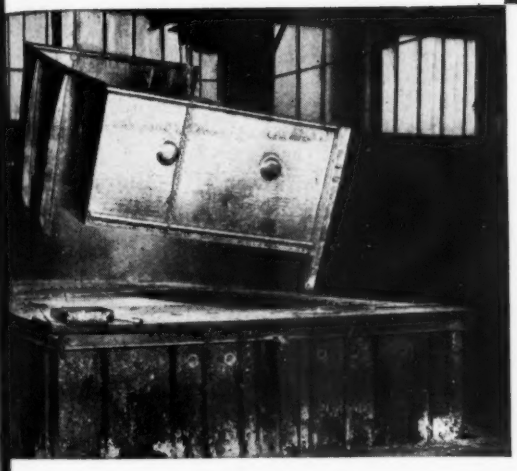
The range of electric immersion heaters for process liquid heating, manufactured under the name Vitreosil by **The Thermal Syndicate Ltd.**, Wallsend, Northumberland, is widely known and applied for the heating of pickling and electroplating solutions. The latest version of these heaters is now available in demountable form with rubber or PVC caps. The PVC caps have been developed for use in solutions involving chromic acid which has been found to attack the normal type of rubber cap. Hitherto these immersion heaters have not been suitable for use in alkaline solutions, and a new development in this connexion has been the replacement of the normal fused silica sheath by one of aluminous porcelain ware in order to provide a heater suitable for use in alkaline environments. These new sheaths (Fig. 5) although showing considerable promise are still under test and will not be marketed until they have passed successfully through the development stage.

The square-wave polarograph shown by **Mervyn Instruments**, St. Johns, Woking, Surrey, can now in its latest form, monitor continuously the concentrations of cyanide and thiocyanate in trade effluents and thus permits the adoption of automatic control for effluent purification plants. The increased sensitivity of the instrument allows the detection of lead, arsenic, copper, etc., at concentrations as low as one part per 1,000 million.

A surface-finish interference microscope was one of the exhibits of **C. Baker of Holborn Ltd.**; very compact in design due to the Mireau optical system, this instrument can be used for metallic and non-metallic surfaces. A camera is available if required.

An automatic grinding and polishing machine developed in collaboration with several metallurgical research associations was exhibited by **R. and J. Beck Ltd.** This equipment provides facilities for the rapid production of polished metal samples for microscopical examination. The specimen holder for one or three specimens is reciprocated automatically relative to the abrasive disc, and the speed of the main spindle is adjustable.

NOT TOO BIG



For Hot Galvanizing

Hot galvanizing is a remarkably versatile rust-proofing process. For buckets, tanks, wire, window frames, bins and countless other items of everyday life, its uses are well known. But few realize how large are many of the articles hot galvanized. Three-storey building stanchions, 2,000-gallon cylinders, road tankers—these things hot galvanizing takes in its stride. But that is not all. Much larger objects are also given long term protection by hot galvanizing the component parts separately and assembling them afterwards. Take the railway goods waggon (bottom left). Measuring 17′ 6″ long by 8′ 6″ wide, this wagon is designed for dipping in four parts—an underframe and three body-sections—which are later bolted together. For T.V. masts the same technique is used. Like all B.B.C. television masts, that at Sutton Coldfield (top right) is built up of hundreds of pre-galvanized sections to ensure the longest possible life without attention. With suitable designing, the sky is the limit!

ot Dip Galvanizers Association

The Hot Dip Galvanizers Association, a non-trading body, welcomes enquiries. Write to 34 Berkeley Square, London, W.1 Tel. Grosvenor 6636

Member of the Zinc Development Association

LARGE-SCALE TUMBLING OF JET-ENGINE COMPONENTS

TO speed production of complex stainless steel jet engine components, specially designed and built tumbling machines are being used at the Des Moines (Iowa) plant of the Solar Aircraft Co. for finishing precision parts and assemblies with large dimensions.

The new machines (which are said to reduce tumbling time 75 per cent.) look like large rotary laundry washers. Each has a drum about 6 ft. long and 4 ft. in diameter, plus a central shaft which can hold as many as 14 large vane and shroud assemblies for new-type J-57 turbojet engines.

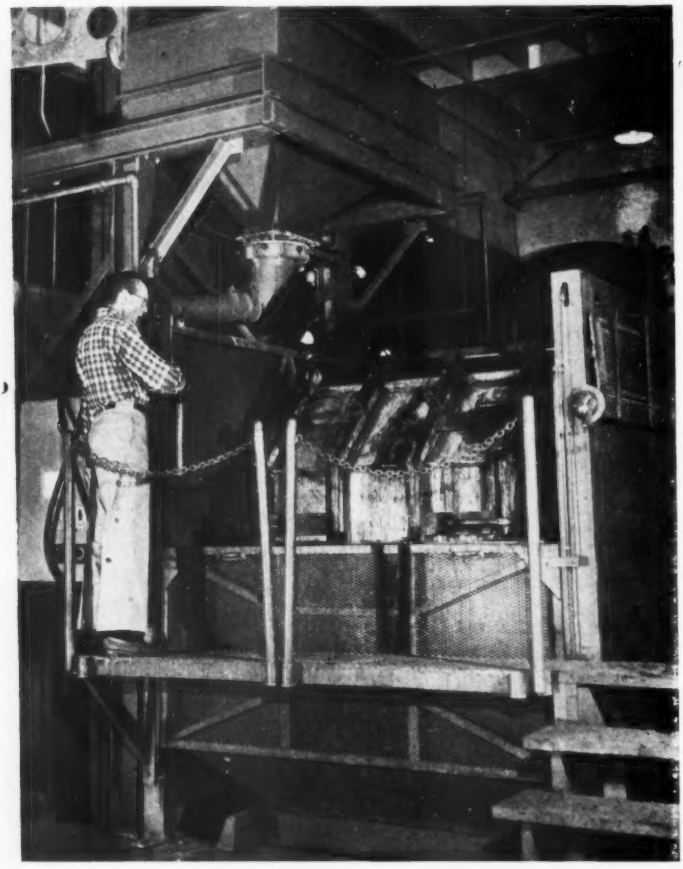
Within the machine, components are rigidly held by the central shaft when low-speed barrel rotations take place.

The barrel contains a two-ton abrasive charge of aluminium oxide chips and pieces of mild steel wire dispersed in water; and as it revolves, this charge hones and polishes sharp edges and nicks, wearing them smooth and clean.

Vane and shroud units finished in the new machines comprise inner and outer rings connected by as many as 160 airfoils.

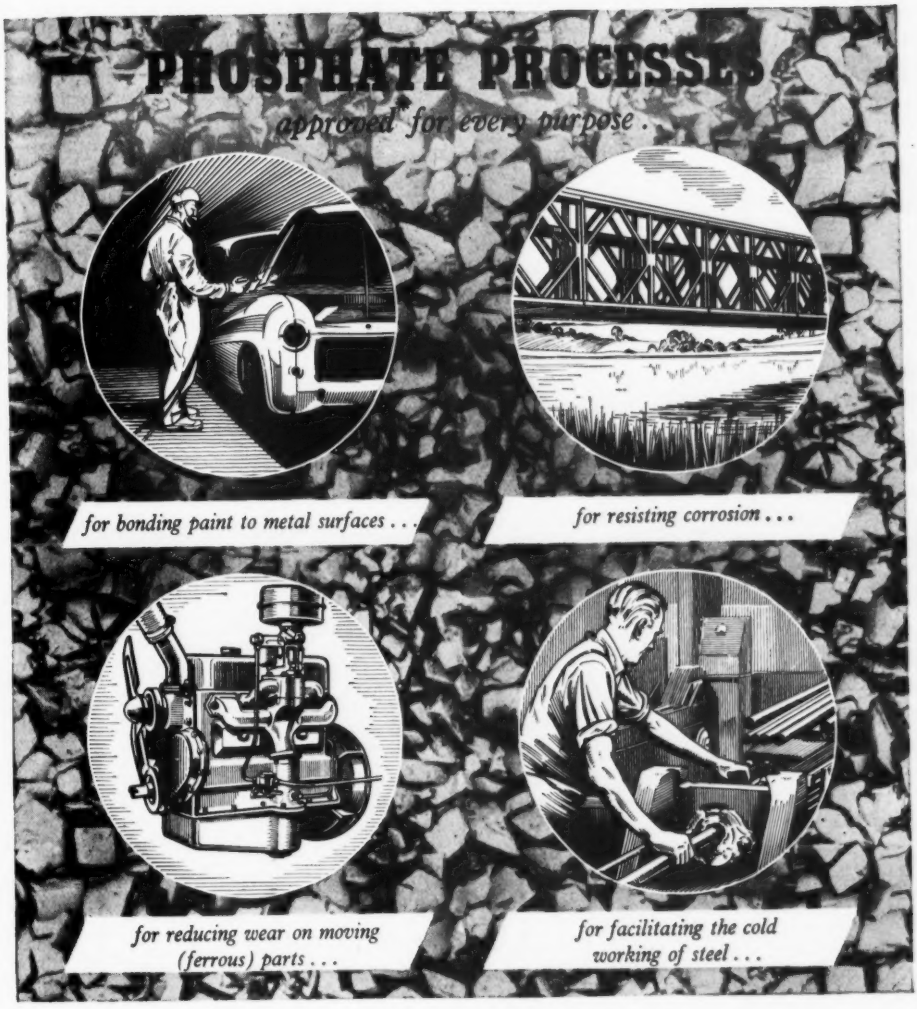
Operated in conjunction with the tumbling units is a special conveyor system which hauls abrasive materials to an overhead hopper, whence the mixture is gravity-fed into a given tumbling barrel each time a new load of parts has been jigged.

Many of Solar's stainless steel components were previously deburred by using hand air grinders, automatic vapour-blast units, and automatic wash cabinets. Inspections after these operations often revealed remaining nicks or sharp edges, which necessitated additional honing and polishing after an initial finishing cycle of one hour.



The new tumblers completely eliminate hand deburring, vapour blasting, and wash operations. In addition, they are said to minimize the possibility of damage to precision-built metal parts.

The photograph on the left shows the tumbling machine specially designed and built for the Solar Aircraft Company. Components for jet engines, in stainless steel, are processed in this equipment which holds a 2-ton abrasive charge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Latest Developments in PLANT, PROCESSES AND EQUIPMENT

Scratch Tester

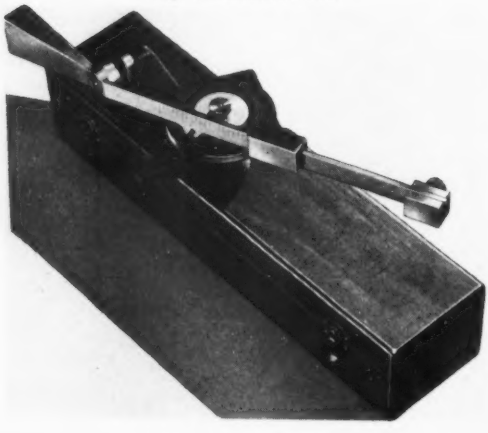
IN ADDITION to the Abraser equipment for testing the abrasion resistance of surfaces described in a recent issue of this Journal, the Taber Instrument Corporation, also manufacture a shear hardness and diamond scratch tester for plastic and organic coatings. This instrument (Fig. 1) is designed for testing the quality of plastic materials and plastic coated surfaces to resist scratches, digs, scrapes and similar physical damage not classed as normal wear. The instrument comprises a finely balanced scale beam to which is attached a precision-ground contour shear tool. This tool has an extremely finely-ground cutting edge lapped to a 25 mm. radius with a 30 deg. clearance and it is set at a 22° shear angle in relation to the motion of the specimen. The tool is designed to cut a groove up to 0·1 in. wide, the width being dependent on the depth of cut.

A sliding weight mounted on the graduated beam can apply a load varying from 0 to 500 gm. by 10 gm. increments. An extra weight can be attached to the end of the beam to bring the total load up to 1,000 gm.

In using the instrument for test purposes a series of trial cuts are made with increasing loads until a groove of the specified width is obtained. The groove should lie between 0·06 and 0·1 in. and for accurate comparisons a certain width of groove should be standardized for similar materials.

In addition to the cutting tool, two types of diamond tool are also available for carrying out scratch tests.

Fig. 1—Scratch Tester



The Taber shear hardness and diamond scratch tester is available in this country from Funditor Ltd., 3 Woodbridge Street, London, E.C.1.

Molybdenum-Disulphide Grease

AFTER much research the Molytone family of greases has been launched by Rocol Ltd., Ibex House, Minories, London, E.C.3, in the United Kingdom, Europe, the Commonwealth and Latin America.

Molytone grease is a combination of a Ben'one grease and molybdenum disulphide possessing many special properties. The temperature-resistance enables machinery to be operated continuously at as high a temperature as 450° F, which is 50° F over what was formerly possible. For short periods lubrication continues satisfactorily at even higher temperatures.

The pressure resistance gives positive lubrication at 100,000 lb. per sq. in. working pressure. Thus high-pressure working conditions, which would normally cause wear, sticking, or seizure, may be countered.

The adhesion to metals is so superior that starved conditions of lubrication can be tolerated without damage to bearings. There is no melting point. The grease can be used in conditions up to the point of inflammability of the basic oil. The excellent water-repellent property allows the grease to be used in high-pressure steam atmospheres and other wet conditions without impairing its lubricating properties.

There is no need to install a special lubricating system, existing grease cups and grease guns are quite satisfactory for the application of Molytone grease, which is of the soft consistency of normal ball- and roller-bearing grease.

25-oz. Air Drill

A NEW light-weight air-powered drill, the CP.3008, is now being produced by the Consolidated Pneumatic Tool Co. Ltd., 232 Dawes Road, London, S.W.6. This tool weighs less than 1½ lb. and is under 6 in. in length.

The drill, powered by a "Power Vane" type motor, is fitted with a Jacob's ¼-in. chuck.

A chuck guard is included as standard equipment and clamp-on attachments may be used with the 1½-in. outside diameter of the tool.

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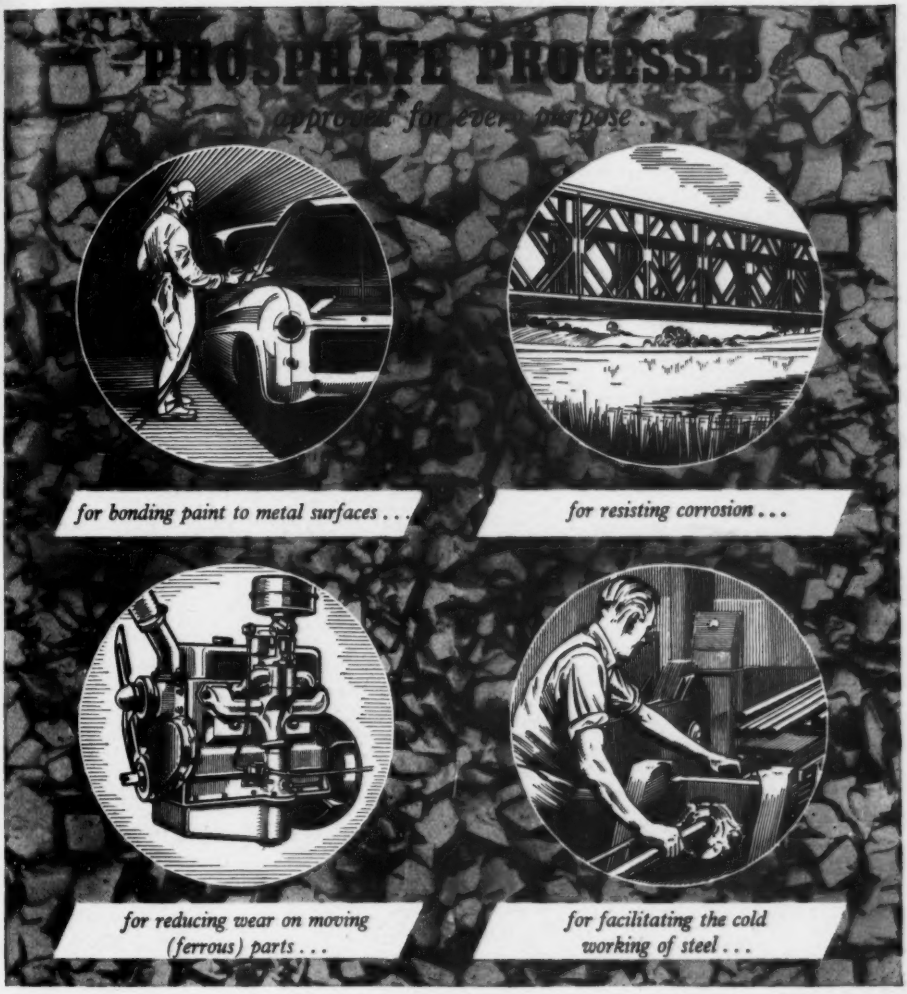
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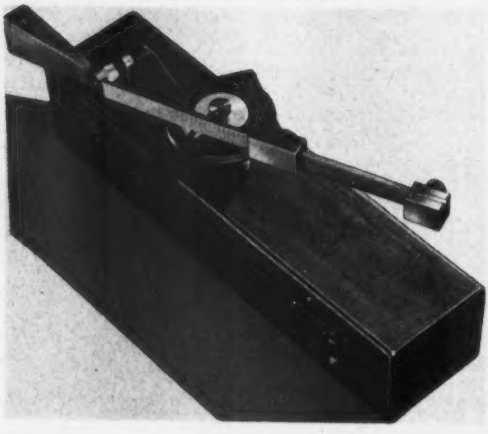

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